STRAIN AGING AND

DELAYED FAILURE IN

HIGH-STRENGTH STEELS

TECHNICAL DOCUMENTARY REPORT No. ASD-TDR-62-968

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DIRECTORATE OF MATERIALS AND PROCESSES
AERONAUTICAL SYSTEMS DIVISION
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WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Project 7351, Task 73521

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FOREWORD

This report was prepared by TAPCO, a division of Thompson Ramo Wooldridge Inc., under USAF Contract No. AF 33(657)-7512. The contract was initiated under Project No. 2(8-7351) and Task No. 73521. The project was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, with Lt. Robert T. Ault acting as project engineer.
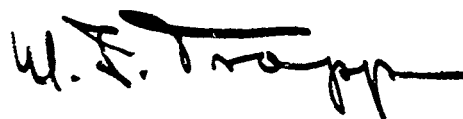
This report covers the period from October 15, 1961, to October 14, 1962.

ABSTRACT

Spontaneous strain aging which occurs during tensile testing was examined for several high-strength steels. The results of smooth and notch tensile tests indicated that significant strain aging effects occurred in most high-strength steels in the 300°F to 800°F temperature range and this behavior was analogous to "blue brittleness" in mild steels.

Constant load, stress rupture tests were conducted on the steels to determine the possible relationship between strain-aging embrittlement and delayed failure. Only the 300 M steel tested at 400°F exhibited an appreciable degree of delayed failure. This embrittlement, was extremely sensitive to test environment and was eliminated when tests were conducted in argon. Although strain aging was not a sufficient condition to initiate delayed failure, it appeared to increase the severity of the environmental effects in the particular range where sufficient interstitial mobility existed.

This technical documentary report has been reviewed and is approved.

W. J. TRAPP
Chief, Strength & Dynamics Branch
Metals & Ceramics Laboratory
Directorate of Materials & Processe

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (Continued)

I INTRODUCTION

The need for airborne components with very high strength-to-weight ratios has generated many unique problems for the materials engineer. These problem areas involve not only the selection of meaningful design strength parameters, but also the consideration of certain types of embrittlements which may limit the performance capacity of these high-strength materials. The irreversible embrittlement produced after tempering in the 500°F to 600°F temperature range (1)* and the time dependent failures due to hydrogen (2) or mildly corrosive environments (3, 4, 5) are well-known examples where the load-carrying ability of a structure can be significantly reduced by embrittlements.

The embrittlement due to strain aging in mild steels is well recognized and has been frequently studied (6, 7, 8). In the case of high-strength steel, however, this phenomenon has only recently received attention and relatively little data exist on the decrease in notch strength which is produced by this factor. Strain aging is generally defined as a time-dependent change in properties that results from a sequence of plastic strain and aging. Under certain conditions of strain rate and test temperature, strain aging can occur spontaneously during a normal tensile test and result in ductility minima and in most cases strength maxima. In mild steels this strain aging process, which occurs during conventional tensile testing, is often referred to as "blue brittleness" (8).

In high-strength steels the sequence of tensile straining followed by aging below the original tempering temperature has been investigated as a method for increasing the smooth strength properties of a material. Recent work with the low alloy martensitic steels (4340) and the hot-work die steels (H-11) has indicated that a significant increase in tensile and yield strength can be produced by this mechanical-thermal treatment (9, 10, 11).

The property variations which have been observed as a result of the straining plus aging sequence have been attributed to a change in the carbide morphology during the aging treatment. The phenomenon of spontaneous strain aging during a tensile test (blue brittleness) has not been systematically evaluated in the high-strength steels. In these materials the major interest has been in the improvement of the transition temperature as measured by a sharply-notched specimen, and relatively little data are available on properties in the super-transition temperature regions where strain aging may be present. In addition, the high-strength steels do not generally exhibit yield point effects and therefore the simple interactions between dislocations and interstitial atoms (8) which are employed to describe strain aging in mild steels are not believed to play a dominant role in the mechanical properties of the ultra-high-strength steels.

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*   Numbers in parentheses pertain to references in the Bibliography.

Recent data by Srawley and Beachem (12) and Preston and Morrison (13) have indicated that a minimum in the notch tensile strength occurs in the low alloy martensitic high-strength steels when they are tested in the super-transition temperature range. Since the temperature at which this decrease in notch properties takes place is comparable to the region where "blue brittleness" occurs in mild steels, the question arises as to whether spontaneous strain aging effects are present to a significant degree in high-strength steels. There is considerable current interest in the mechanical properties of materials in this super-transition temperature region since this is the temperature spectrum under consideration for the supersonic transport. Certain pressure vessel applications may also involve temperatures up to 500°F and any decrease in load-carrying capacity may be a critical consideration at these temperatures which are above the conventional sharp-notch transition temperature.

Since strain aging is a time dependent phenomenon, there is the possibility that this embrittlement will also be operative in constant load tests where the test time is a maximum. Troiano (2) has recently proposed a mechanism for strain aging embrittlement which predicts this effect and which involves the clustering of interstitial atoms at the tip of a crack or dislocation pile-up. The interstitial grouping is believed to lower the cohesive strength of the material and cause crack extension. Although his results are based on work with hydrogenated steels, the possibility exists that carbon and/or nitrogen strain aging can also lead to delayed failure in temperature regions where these interstitials have a mobility similar to hydrogen at room temperature. The high-strength steels with their marginal crack propagation resistance represent materials which may be particularly susceptible to a non-hydrogen induced type of static fatigue.

The purpose of this investigation was to evaluate the magnitude and kinetics of spontaneous strain aging in high-strength steels and determine its possible association with delayed failure under static loads.

II MATERIALS EVALUATED

Four basic types of high-strength steel (see Table I) were selected for the experimental program. All the materials, with the exception of the Ni-Co-Mo maraging type, were conventionally air melted. The maraging steel was received from a commercial consumable electrode, vacuum-melted heat. The 300 M and 4340 steels were selected as representative of the low alloy martensitic high-strength steels while the H-11 was evaluated as a characteristic hot-work die steel. The 17-7 PH and AM 355 were chosen as typical precipitation-hardening stainless steels. The Ni-Co-Mo alloy was included as representative of the recently developed maraging steels. The heat treatments employed for these materials are summarized in Table II. All the austenitizing treatments were performed in neutral salt baths while the tempering was done in an air furnace. No stock was removed from the surface of the material. In the as-received condition the 300 M steel had a slight degree of surface decarburization which produced a hardness difference of approximately $2 R_c$ from the surface to a depth of 0.002". The other steels used in the investigation had no significant decarburization.

III TEST PROCEDURE

Strain aging effects in high-strength steels were studied by conducting smooth and notch tensile tests as a function of loading rate and test temperature. The maxima in ultimate strength or the minima in notch strength* were employed to evaluate the strain aging phenomenon. The smooth tensile specimen geometry is presented in Figure 1, while the notch tensile specimen is shown in Figure 2A. The notch specimen corresponded to the recommendations of the ASTM Committee on Fracture Testing of High-Strength Materials (14) and consisted of a center notch generated by fatigue cracking after heat treatment.

The correspondence between strain aging and delayed failure was evaluated by conducting stress-rupture tests under sustained loads in temperature regions where strain aging occurred in the tensile test. Notch specimens with the geometry shown in Figure 2A were used for 300 M tempered at 600°F because relatively low breaking loads were required. In other materials, the breaking loads exceeded the capacity of the stress-rupture apparatus and the notch specimens shown in Figure 2B were used.

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* Notch strength is defined as the load at fracture divided by the initial cross-section area at the plane of the notch.

3

TABLE I

Nominal Compositions of the Material used in this Investigation

Designation	Thickness	Carbon	Chromium	Nickel	Moly.	Silicon	Titanium	Other	Iron
				Element – Weight Per Cent					
300 M	0.080"	0.40	0.90	1.85	0.40	1.75	--	--	Balance
4340	0.080"	0.40	0.90	1.85	0.40	--	--	--	Balance
H-11	0.080"	0.40	5.0	--	1.30	1.00	--	--	Balance
17-7PH		0.07	17.1	7.20	--	0.40	--	0.15 Al	Balance
AM 355		0.15	15.5	4.25	2.75	0.30	--	0.10 N	Balance
Ni-Co-Mo*	0.080	0.01	--	18.17	4.77	--	0.42	7.64 Co	Balance

* Heat analysis

4

TABLE II

Material Heat Treatment

Material	Hardening Conditions	Tempering Conditions
300M	Normalize 1700°F, salt, air cool Austenitize 1600°F, salt, oil quench	Double Temper - 1 hour cycles 600°F or 1025°F
4340	Austenitize 1550°F, salt, air cool	Double Temper - 1 hour cycles 400°F or 750°F
H-11	Austenitize 1850°F, salt, air cool	Double Temper - 2 hour cycles at 1000°F
17-7PH	Condition 1700°F, salt, air cool Subzero cool 8 hour @ -110°F	Age 3 hours at 950°F (RH 950)
AM 355	Condition 1900°F, salt, air cool Condition 1700°F, salt, air cool Subzero cool 3 hour @ -110°F	Age 3 hours at 850°F (SCT 850)
Ni-Co-Mo Maraging Steel	Austenitize 1500°F, salt, air cool	Age 3 hours at 900°F

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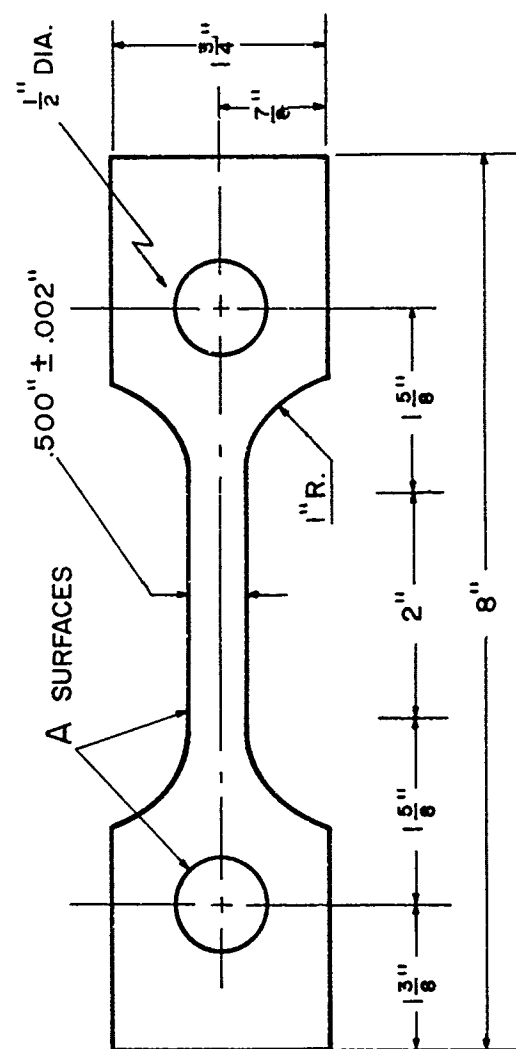
FIG. 1: SMOOTH SHEET SPECIMEN FOR TENSILE TESTS.

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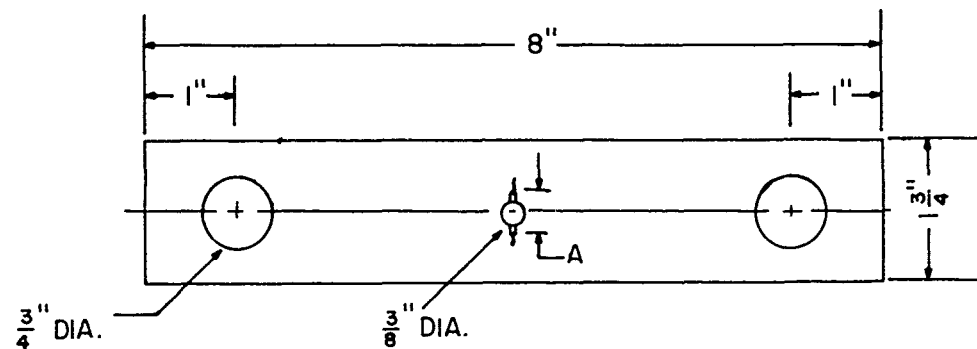
SAWCUT A APPROX. 0.600" EXTENDED BY FATIGUE
PRECRACKING TO 0.700"

FIG. 2A
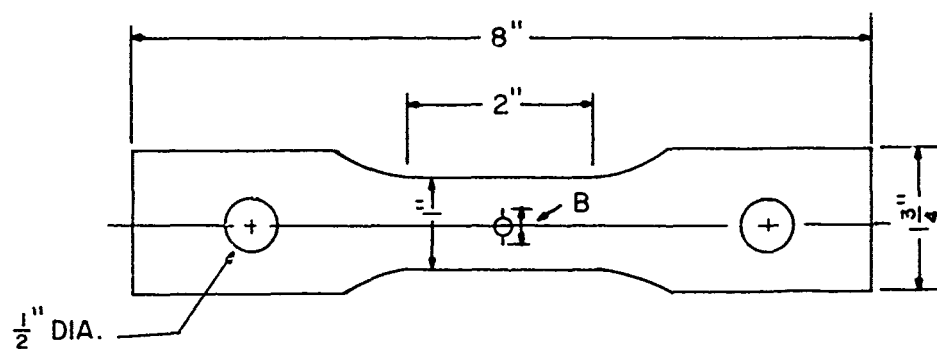
SAWCUT B APPROX. 0.350" EXTENDED BY
FATIGUE PRECRACKING TO 0.400"

FIG. 2B

FIG. 2: GEOMETRY OF NOTCH TENSILE SPECIMENS.

In order to study the kinetics of the slow crack propagation under
sustained load, a resistance bridge was employed. The details of the
resistance technique which have been previously described (15, 16),
involve making the specimen one leg of a Kelvin double bridge and measuring
the increase in resistance which accompanies the crack extension. Although
the majority of sustained-load tests were conducted in an air environment,
in certain instances an argon atmosphere was also employed. Figure 3
illustrates the apparatus used for the tests conducted in a dynamic argon
environment.

IV RESULTS AND DISCUSSION

A. Smooth and Notch Tensile Tests

The results of smooth tensile tests conducted on 300 M (600°F temper)
as a function of test temperature are presented in Figure 4. These data
which were obtained at a crosshead speed of 0.050"/min. indicate that there
is an anomalous increase in the tensile strength at approximately 300°F and
a possible decrease in ductility as measured by percent elongation in
essentially the same range of test temperatures. The results obtained with
300 M notched specimens presented in Figure 5 indicate a very noticeable,
strain rate dependent embrittlement. In accordance with conventional behavior
for a thermally activated process, such as strain aging, the temperature where
embrittlement occurred decreased as the testing time increased (i.e. at de-
creasing strain rates). Figure 6 illustrates a comparable type of notch
embrittlement which occurred in the 300 M tempered at 1025°F. In this case
the region of maximum embrittlement occurred at a slightly higher test
temperature than when the steel was tempered at 600°F.

The results of smooth and notch tensile tests of 4340 steel tempered
at 750°F are shown in Figures 7 and 8. Although the results are similar
to those obtained with the 300 M, the degree of notch embrittlement in the
4340 steel at this strength level was more severe.

The smooth and notch tensile properties of the H-11 die steel are
presented as a function of test temperature in Figure 9. This material,
which was double tempered at 1000°F, exhibited no anomalies in properties
which could suggest significant strain aging effects. It is interesting
to note however, that the notch tensile strength as determined by the
precracked specimens was below the yield strength over the entire range of
test temperatures.

The 17-7 PH stainless steel (see Figure 10) also exhibited no noticeable
maxima or minima in mechanical properties that would be indicative of spon-
taneous strain aging during the test. As shown in Figure 11, the AM 355
precipitation-hardening stainless steel, in agreement with previously
published data (17), exhibited a maxima in tensile strength in the 600°F
to 800°F test temperature range. The peak tensile strength values were
shifted to lower temperatures with decreasing strain rate, indicating that
a strain aging phenomenon was operative. Although the notch tensile tests

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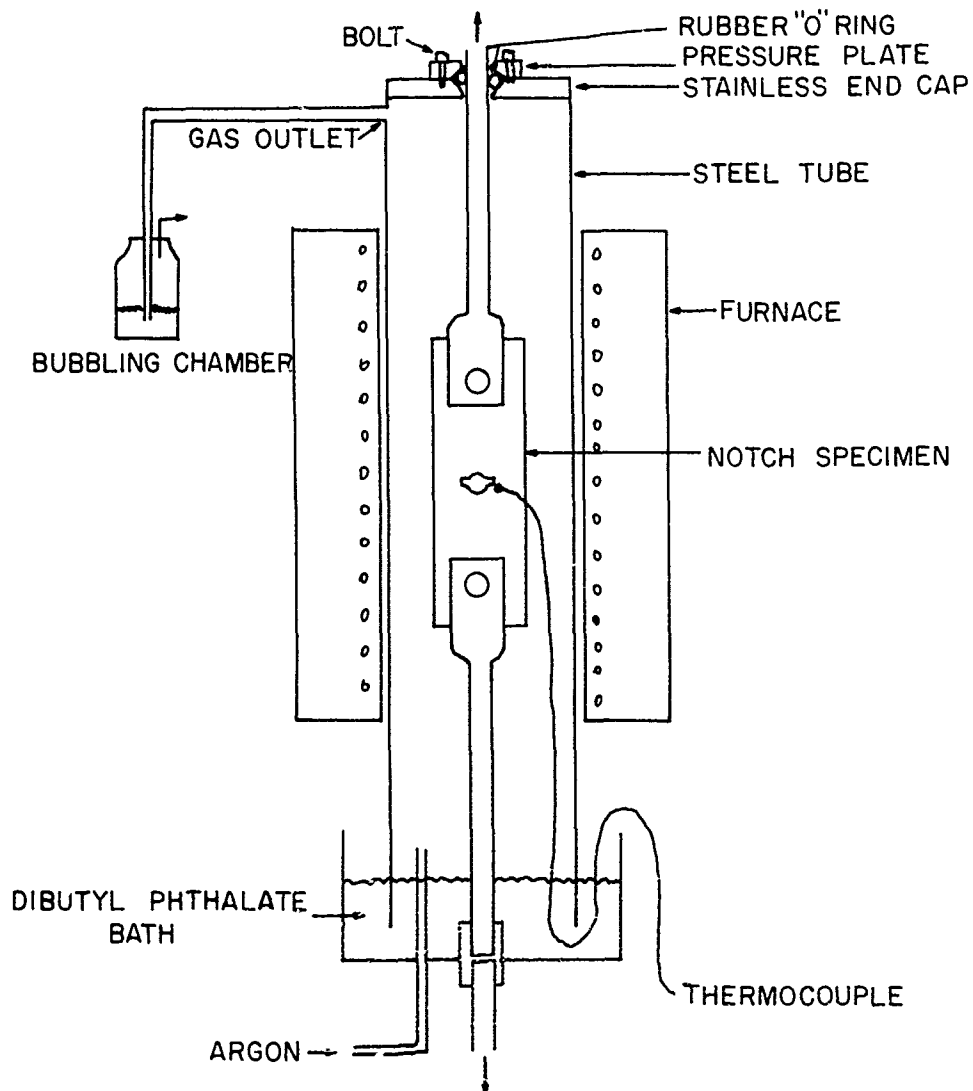
FIG. 3: APPARATUS FOR MECHANICAL TESTING IN AN ARGON ATMOSPHERE.

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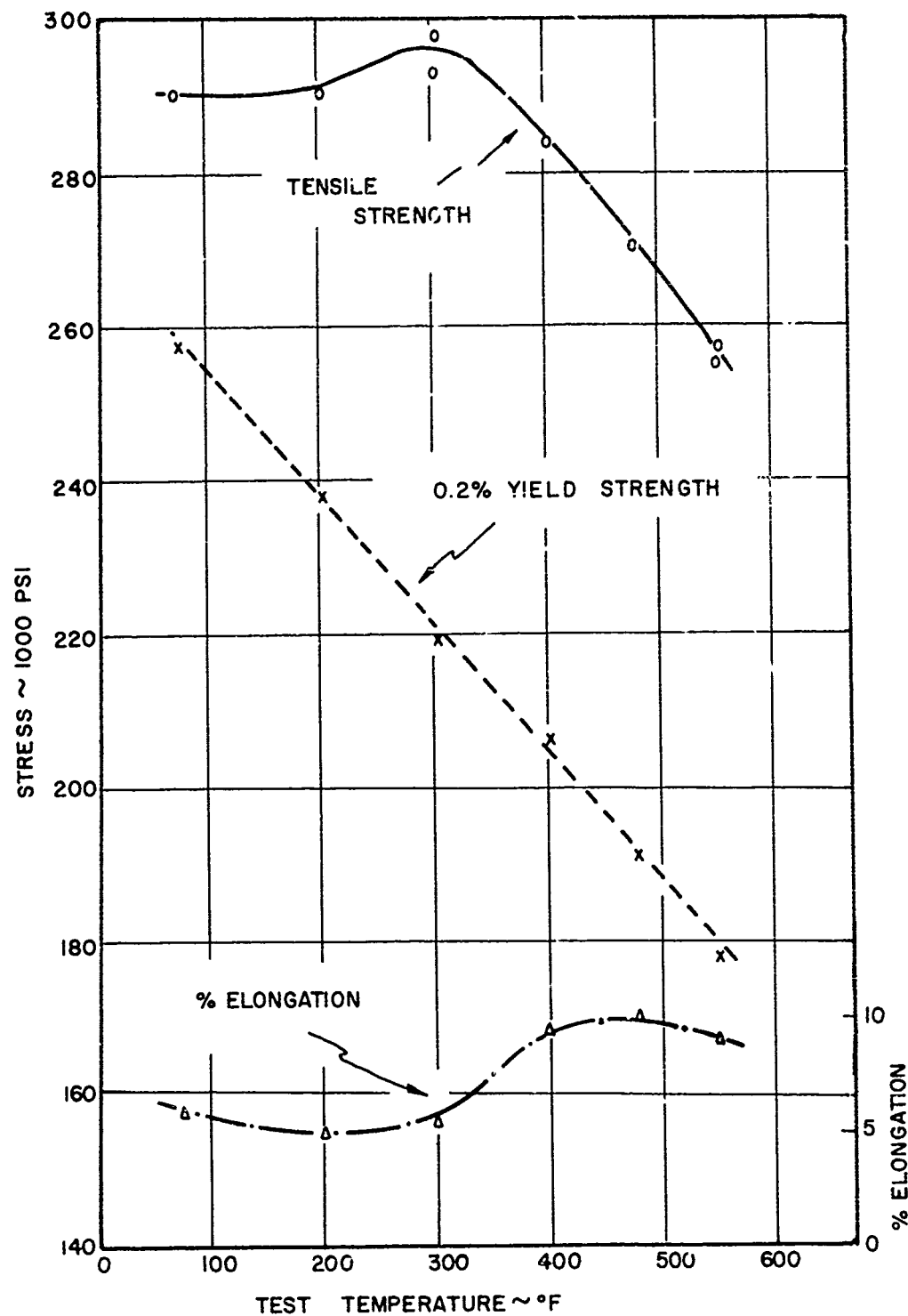
FIG.4: TENSILE PROPERTIES OF 300M STEEL (600°F TEMPER), CROSSHEAD SPEED 0.050"/MIN.

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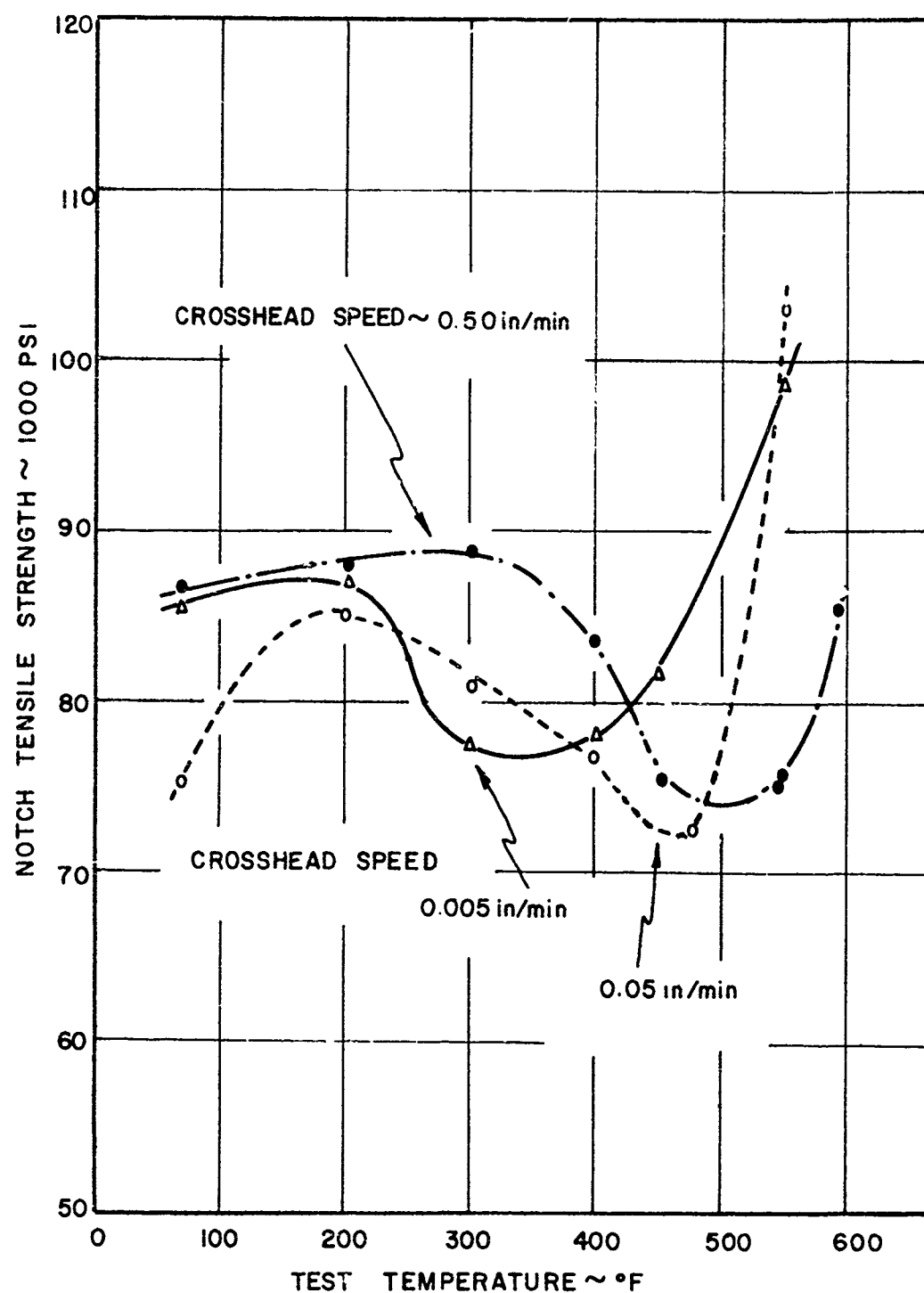
FIG.5: NOTCH TENSILE PROPERTIES OF 300M STEEL, (600°F TEMPER).

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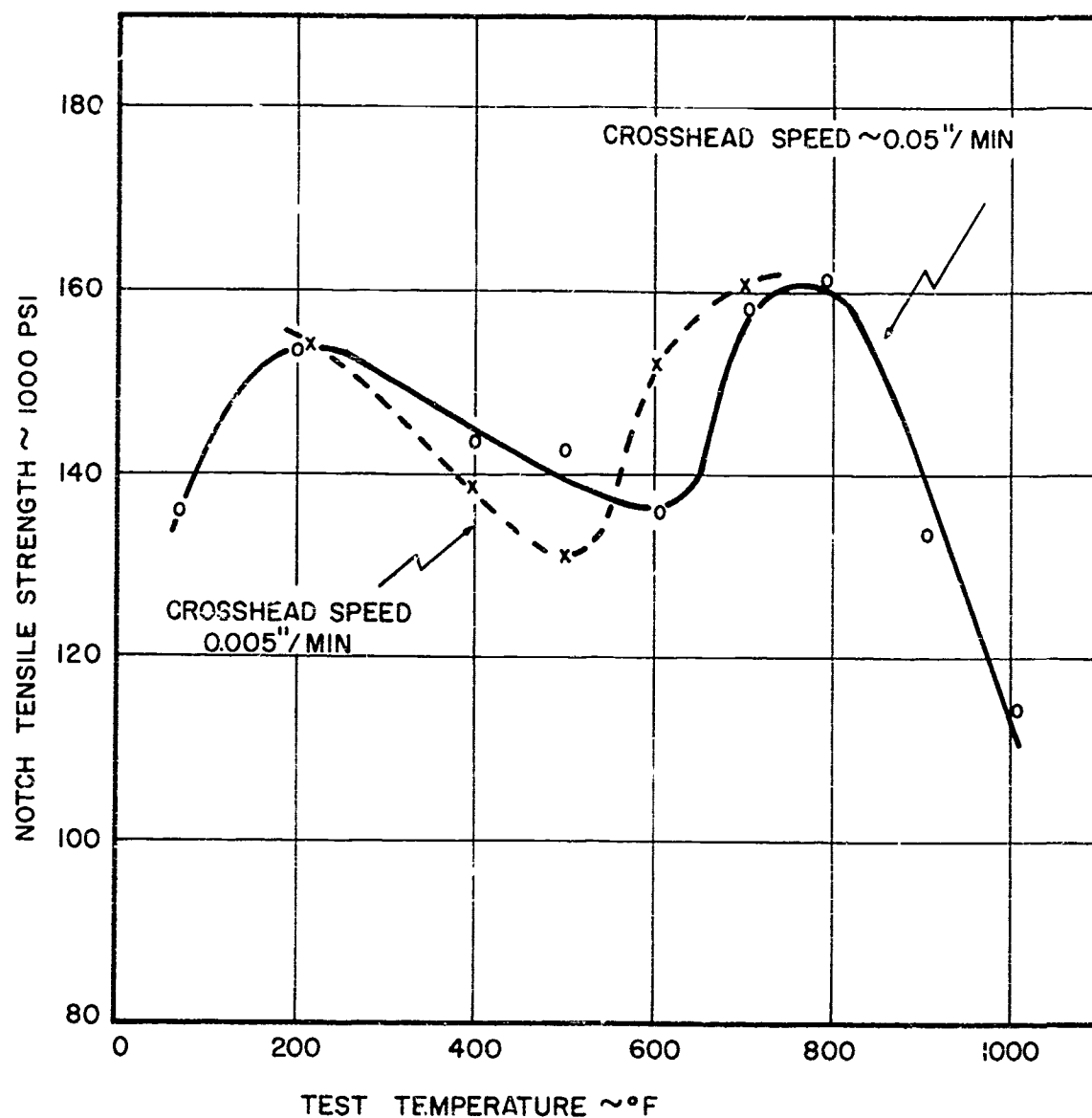
FIG. 6: NOTCH TENSILE STRENGTH OF 300 M STEEL (1025°F TEMPER) AS A FUNCTION OF TEST TEMPERATURE.
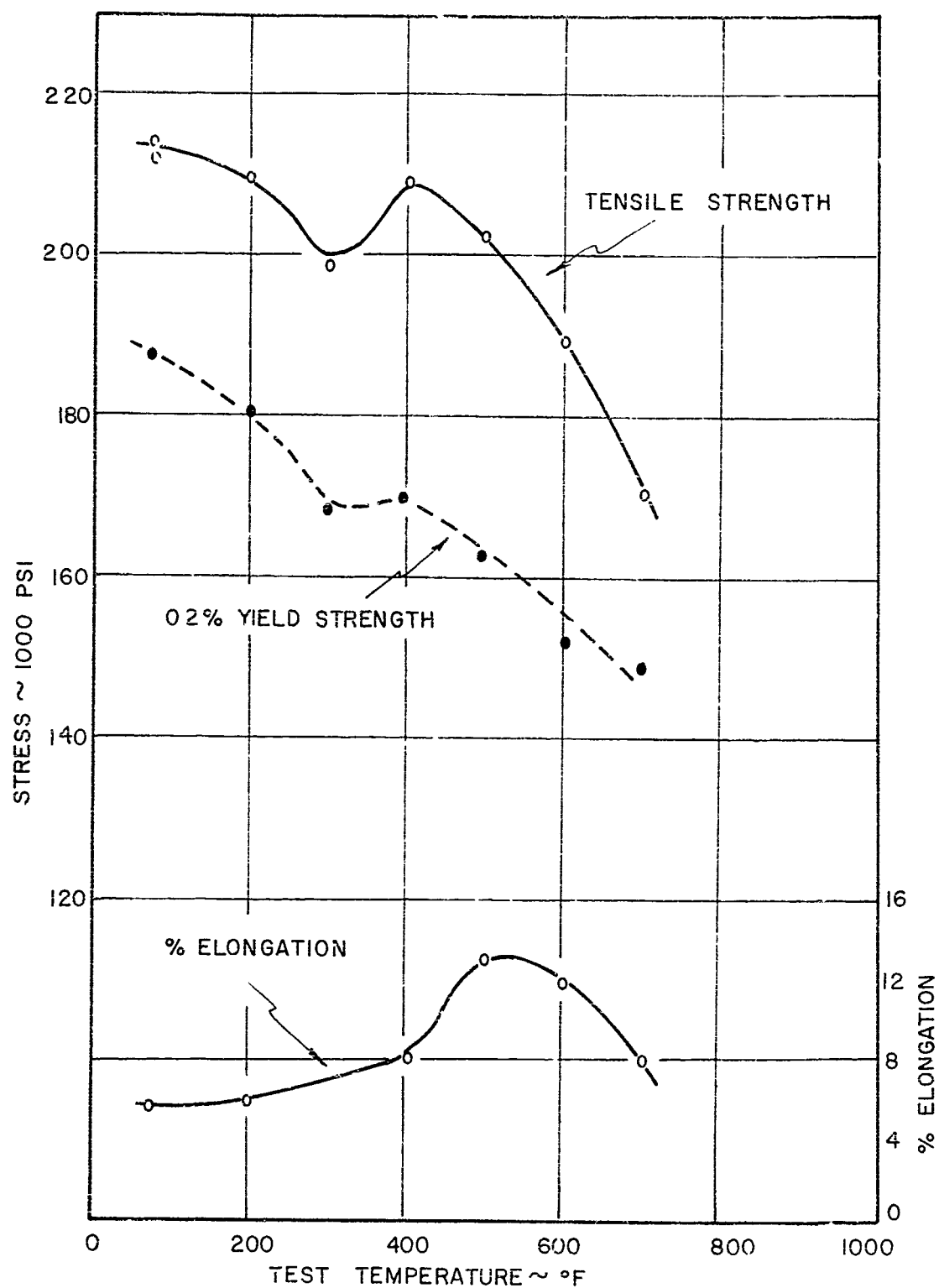
FIG.7: TENSILE PROPERTIES OF 4340 STEEL (750°F TEMPER) AS A FUNCTION OF TEST TEMPERATURE, CROSSHEAD SPEED 0.050"/MIN.

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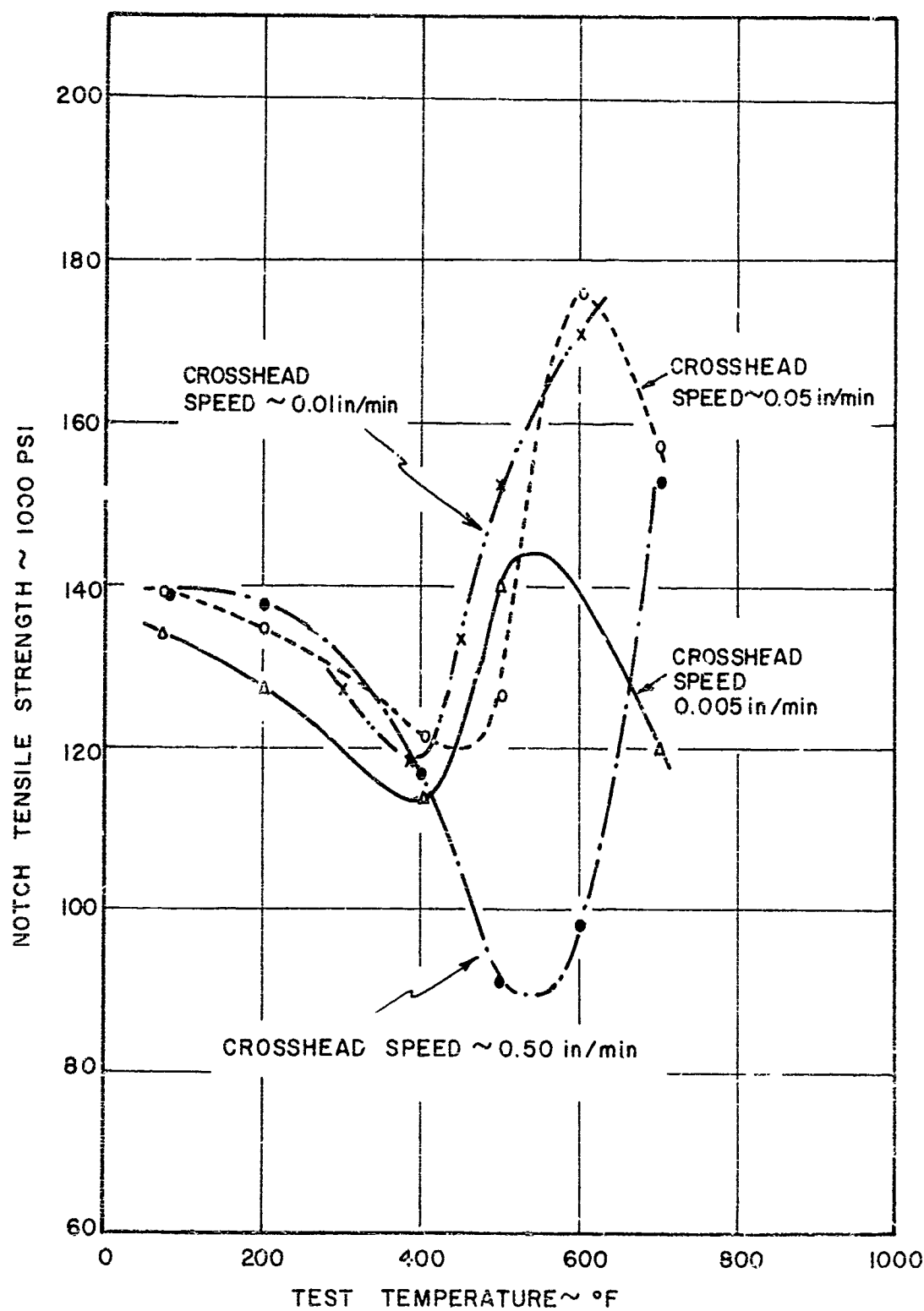
FIG.8: NOTCH TENSILE PROPERTIES OF 4340 STEEL (750°F TEMPER).

14

FIG. 9: TENSILE PROPERTIES OF H-II STEEL, 1000°F
TEMPER, CROSSHEAD SPEED 0.050"/MIN.

FIG.IO: TENSILE PROPERTIES OF 17-7PH STEEL (RH 950)
AS A FUNCTION OF TEST TEMPERATURE. CROSSHEAD
SPEED 0.050"/MIN.

16

FIG.II. TENSILE PROPERTIES OF AM 355 STEEL (SCT 850).

suggest that a minimum in notch properties may be present at temperatures greater than 800°F, the data are inconclusive. Test temperatures greater than 800°F were not employed to avoid the introduction of variations in microstructure which may be produced by testing above the aging temperature used during heat treatment.

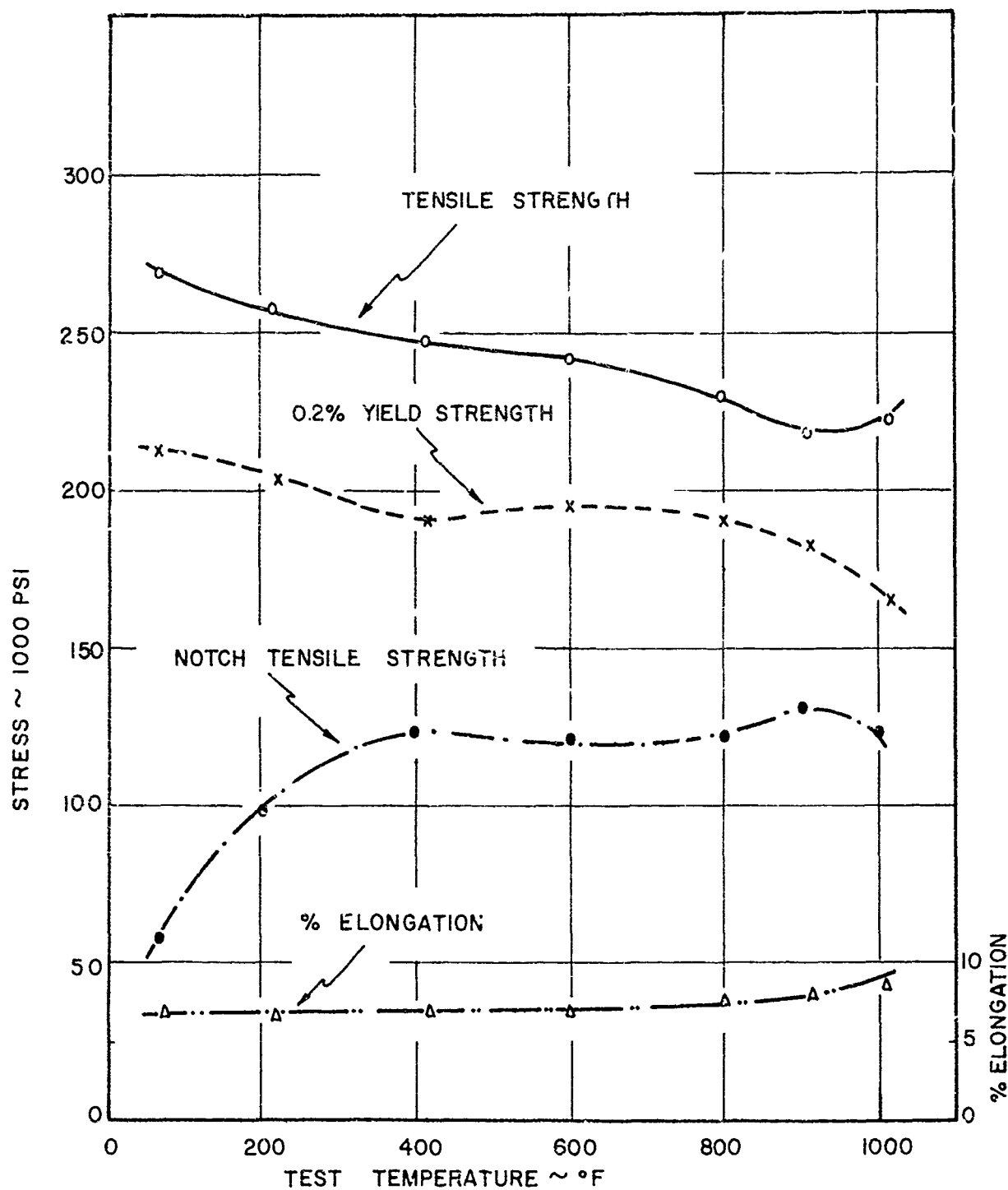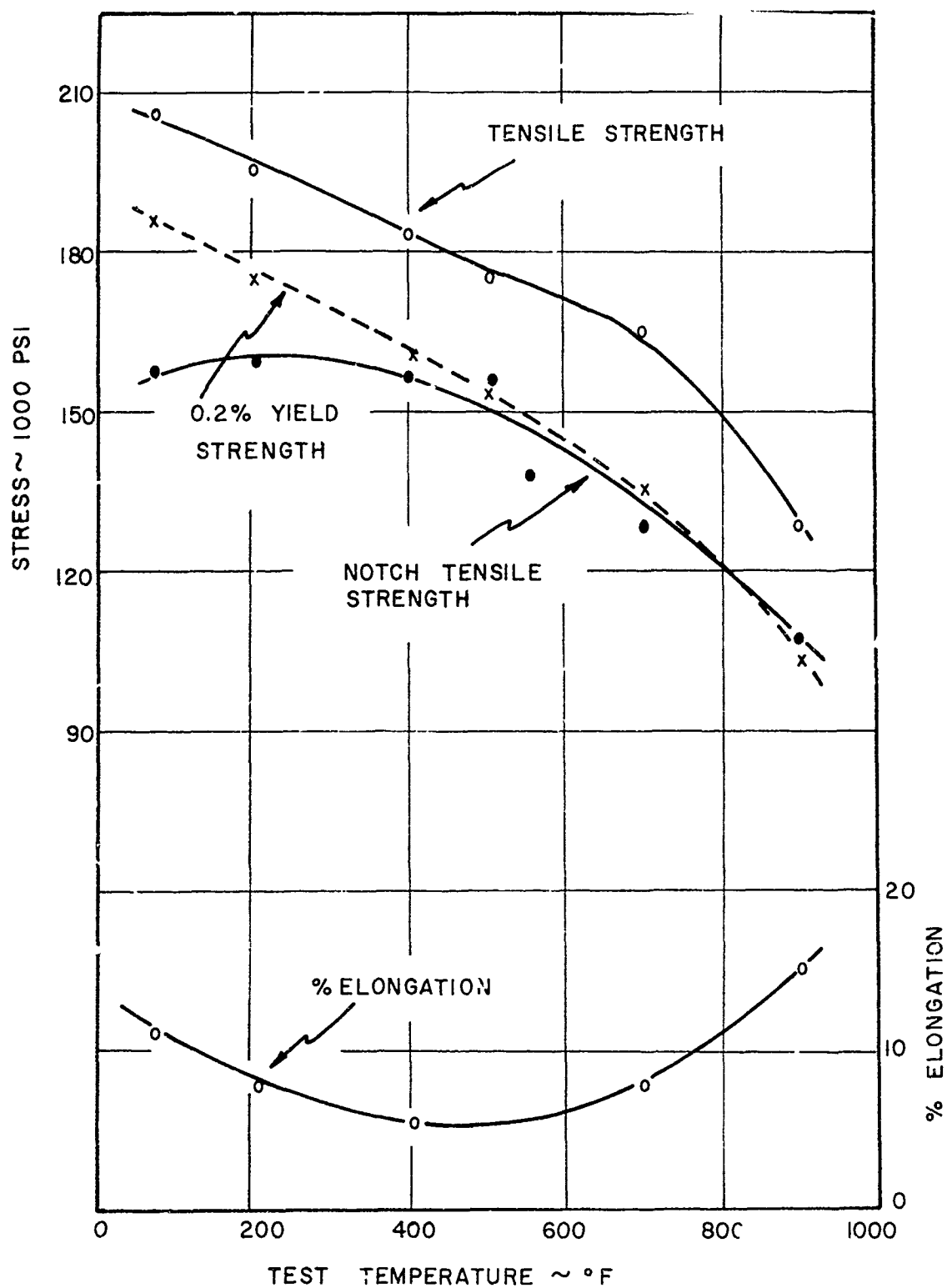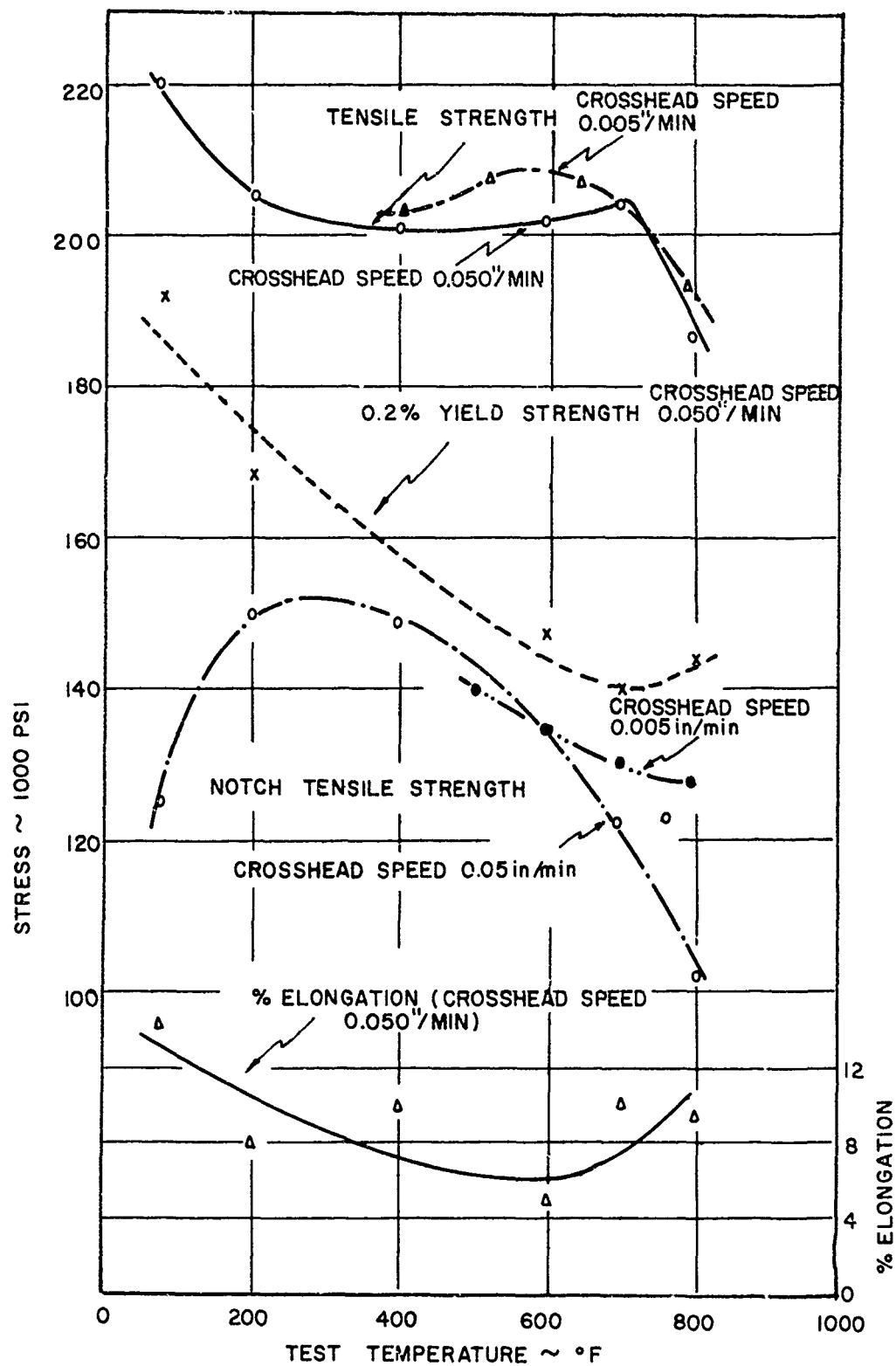The results of tensile tests conducted on the maraging steel are presented in Figure 12. The data obtained from smooth tensile specimens indicate that a peak in both tensile and yield strength occurred at approximately 650°F when a crosshead speed of 0.050"/min. was used. Definite minima in notch strength values were observed in approximately the same region of test temperature where the increase in smooth strength occurred. The minima in notch tensile strength were shifted to slightly lower temperatures when the strain rate was decreased.

The influence of strain aging during the tensile test was observed in varying degrees in the low alloy martensitic, the maraging, and the AM 355 steels. The 17-7 PH and the H-11 steels did not exhibit any strain aging effects for the particular heat treatments investigated. There was a general tendency for the phenomenon to occur at higher test temperatures in steels which were heat treated at higher tempering temperatures. The morphology of the carbide may therefore play a dominant role in the strain aging behavior.

The presence of the strain-aging type embrittlement in high-strength steels raises two important questions:

1)   What influence will this strain aging have in engineering applications?

2)   What is the mechanism producing the strain aging behavior?

The importance of the strain-aging embrittlement in determining the performance capabilities of structural components is difficult to generally evaluate since it is dependent on the particular application. In cases, such as AM 355, Ni-Co-Mo maraging steel, and 300 M tempered at 1025°F, the basic crack propagation resistance of the material as measured by conventional room temperature tests was relatively high. In these steels the strain aging phenomenon produced only a slight degradation in notch properties and therefore would not be expected to significantly influence component reliability. The strain aging behavior present in steels such as 4340 tempered at 750°F and 300 M tempered at 600°F produced a more pronounced decrease in notch strength and under these conditions the embrittlement could be an important consideration.

Strain-aging effects in tensile tests may be indicative of susceptibility to delayed failure under constant load. This type of static fatigue can be a particularly perplexing engineering problem since the embrittlement may not be apparent in conventional tensile tests and only occur under the proper sequence of time, temperature and applied stress. Although the classical example of static fatigue of high-strength steels occurs with hydrogen,
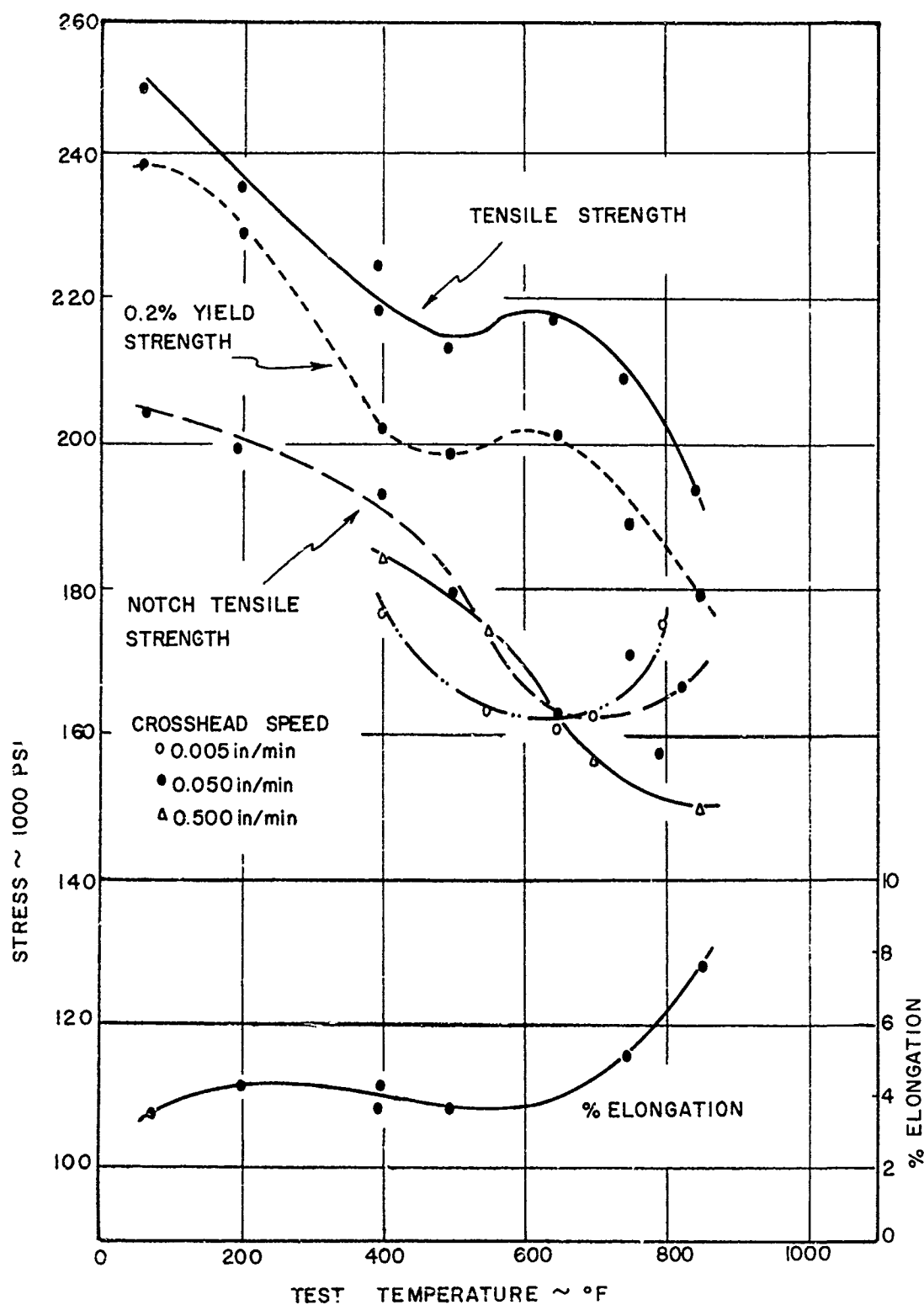
18

FIG.12: TENSILE PROPERTIES OF 18% NICKEL MARAGING STEEL.

19

there is a theory which predicts that comparable effects could be produced by other interstitials (2). In fact, there have been instances of non-hydrogen induced delayed failure which have been attributed to carbon and/or nitrogen (18). In addition, extensive sustained load tests conducted on steels for bolt applications at temperatures in the 900 - 1200°F range have indicated a time-temperature dependent notch embrittlement (19). The question arises as to whether the strain aging behavior observed in this investigation can produce a significant embrittlement under conditions of sustained load, where the time for interstitial movement is maximized.

B. Failure of High-Strength Steels Under Sustained Loads

The results obtained from stress rupture tests conducted at 300°F, 400°F, and 550°F with 300 M high-strength steel tempered at 600°F are shown in Figure 13. In order to compare the results for different temperatures, the applied stress was normalized by dividing it by the conventional notch tensile strength obtained at the particular test temperature and with a crosshead speed of 0.050"/min. (see Figure 5). The specimens tested at 400°F, which is in the temperature region where strain aging was prominent in the tensile test, failed in relatively short times at applied stress values as low as 0.6 the normal notch tensile strength. This rather large range of delayed failure was not present in tests conducted at 300°F and 550°F. The fact that failures did not occur at 550°F indicates that the fractures at 400°F could not be attributed to conventional creep processes. It is also very improbable that the failures could be due to hydrogen since the steel was in an unhydrogenated condition, and hydrogen-induced delayed failures are not present at temperatures above 300°F (20).

Sustained load tests conducted on 300 M specimens tempered at 1025°F did not show any failure in 200 hours at temperatures below 800°F and at applied stress levels which were 90% of the normal notch tensile strength. The static fatigue curves obtained from notch tests at 800°F and 900°F are shown in Figure 14. In this series of tests the failures appear to be due to conventional creep behavior.

Stress rupture tests conducted on notch specimens of the 18% nickel-maraging steel are shown in Figure 15. Once again no appreciable region of delayed failure was present which could be attributed to non-creep type processes. Sustained load tests on 4340 steel tempered at 750°F, and AM 355, showed no delayed failure in 200 hours at an applied stress of 0.9 times the conventional notch strength and in temperature regions where strain aging was experienced.

Since the association between strain aging and delayed failure was only apparent to any appreciable extent in the 300 M steel tempered at 600°F, this material was selected for further evaluation of the delayed failure mechanism. The macroscopic appearance of the fracture surface of a specimen of 300 M steel which failed under static load is shown in Figure 16. The failure pattern consisted of the following segments:

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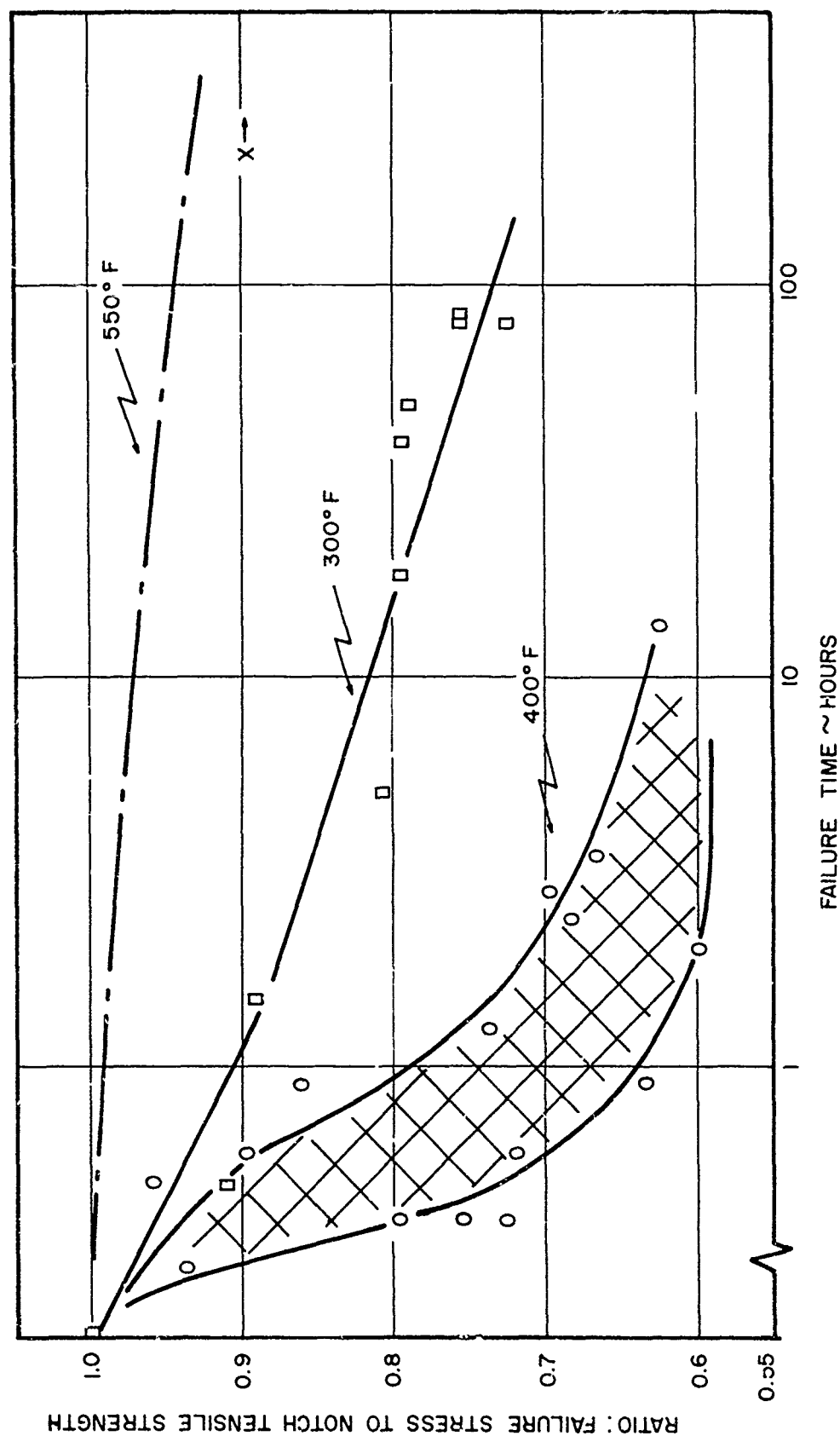
FIG. 13: INFLUENCE OF TEST TEMPERATURE ON THE DELAYED FAILURE CHARACTERISTICS OF 300 M STEEL, (600°F TEMPER), NOTCH TESTS IN AN AIR ENVIRONMENT.

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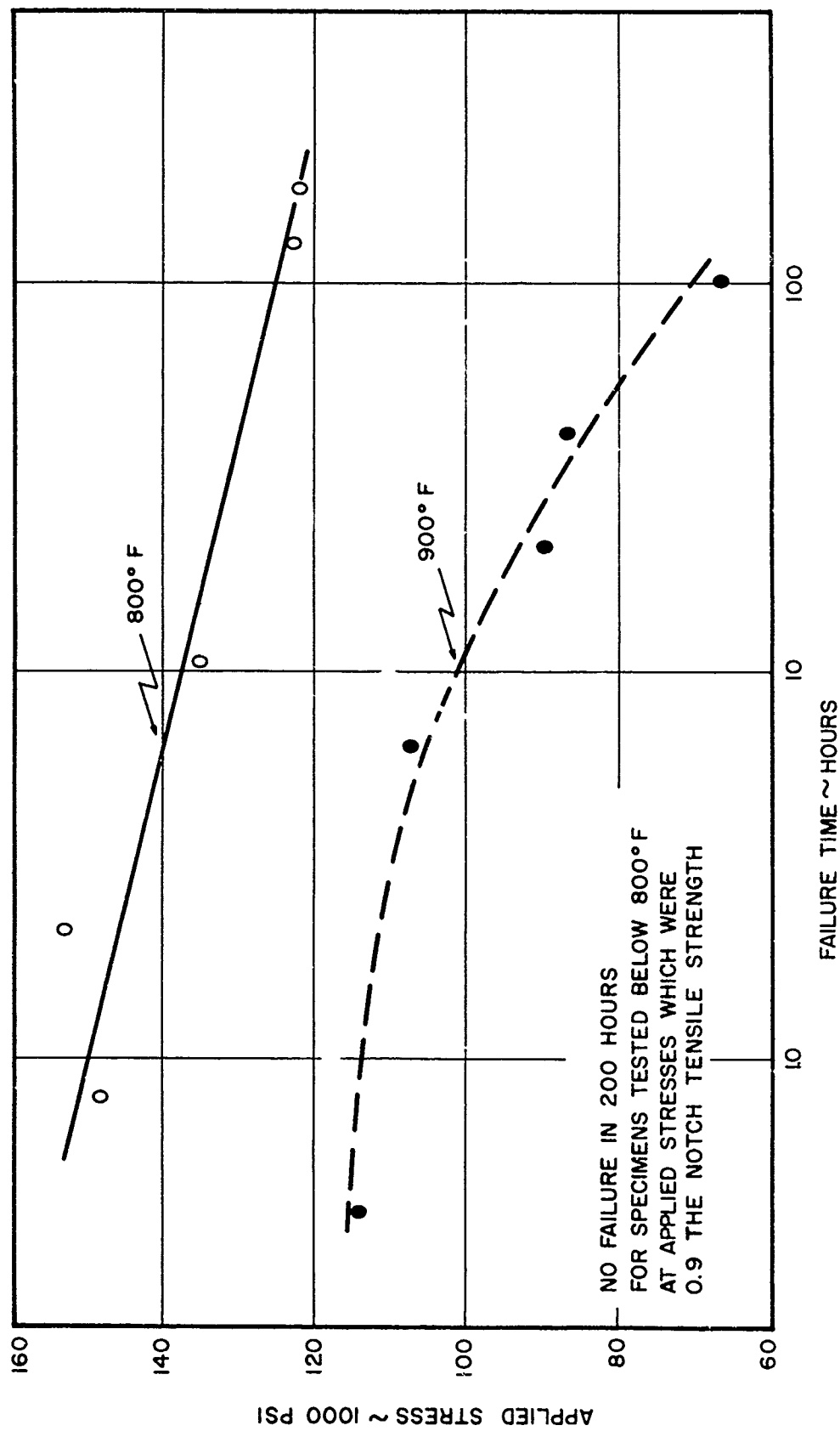
FIG.14: INFLUENCE OF TEST TEMPERATURE ON THE STRESS RUPTURE CHARACTERISTICS OF 300 M STEEL, (1025°F TEMPER) NOTCH SPECIMENS IN AN AIR ENVIRONMENT.

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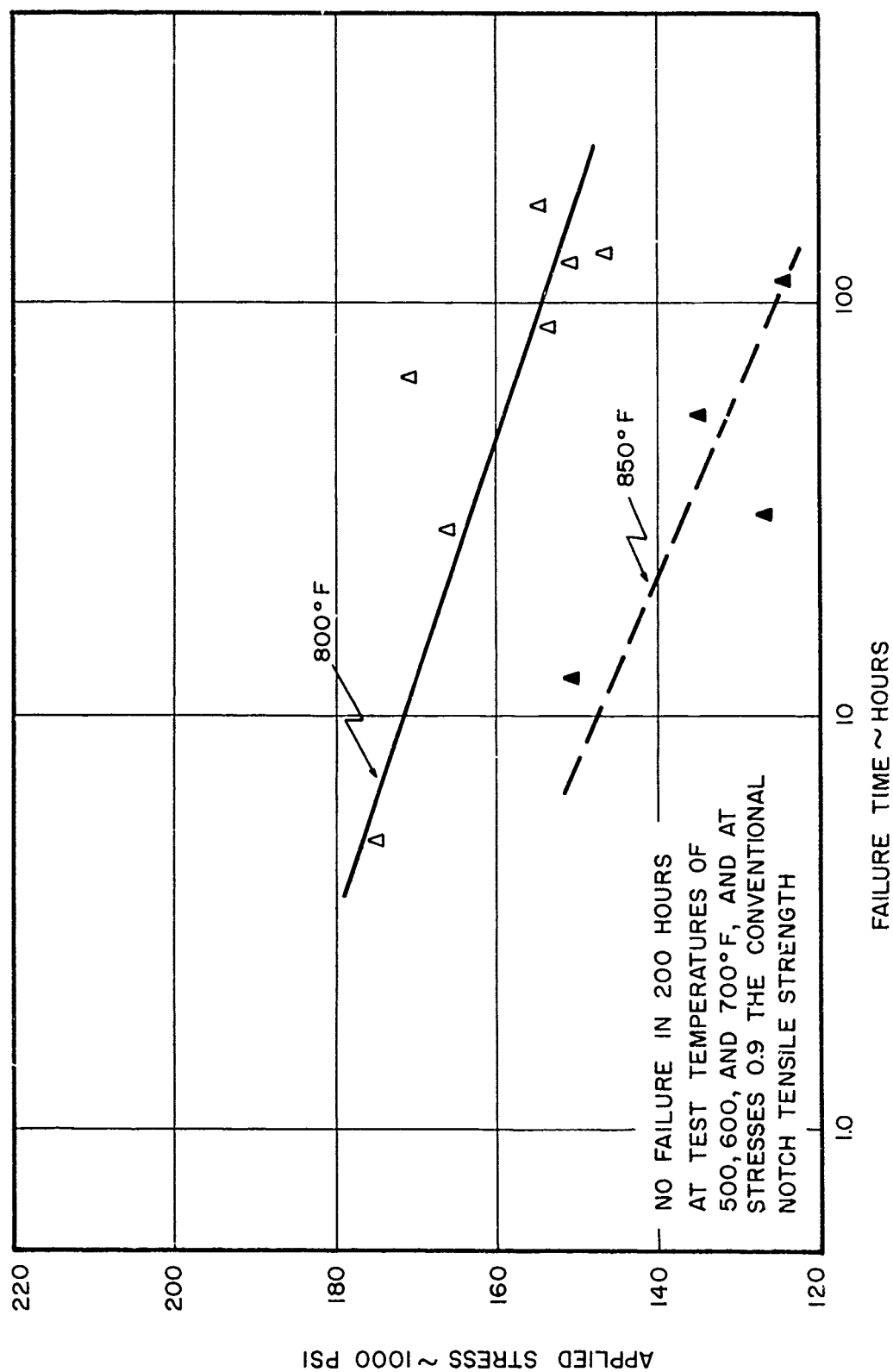
FIG. 15: INFLUENCE OF TEST TEMPERATURE ON THE STRESS RUPTURE CHARACTERISTICS OF 18% NICKEL MARAGING STEEL (240,000 PSI YIELD STRENGTH), NOTCH SPECIMENS IN AN AIR ENVIRONMENT.

23

06378-3

FATIGUE CRACK
CRACK EXTENSION ON INITIAL LOADING

SAWCUT | RAPID CRACK PROPAGATION

INITIAL NOTCH

SLOW CRACK GROWTH
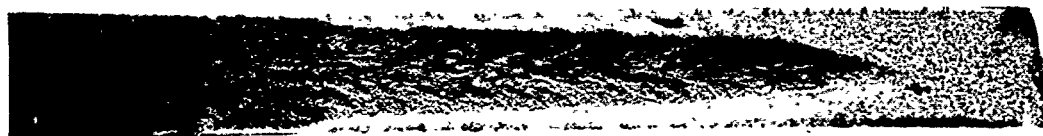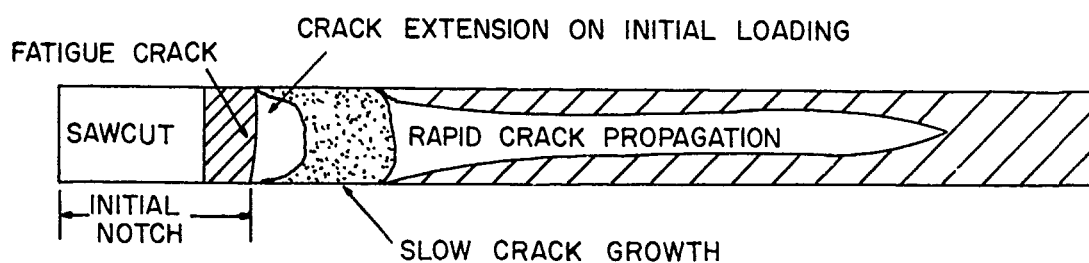
APPLIED STRESS = 64,700 PSI ;   FAILURE TIME = 19.5 HOURS

FIG.16: MACROSCOPIC APPEARANCE (MAG. 10ᵡ) OF SUSTAINED LOAD FAILURES IN 300M STEEL SPECIMEN , (600°F TEMPER) TESTED AT 400°F IN AN AIR ENVIRONMENT.

24

a) Initial notch (sawcut plus fatigue precrack),

b) Crack extension which occurred during the application of a sufficiently high initial load,

c) Slow crack growth which occurred under sustained load,

d) Rapid crack propagation.

The heat tinting which occurred during the test allowed each mode of crack propagation to be readily distinguished. The appearance of the slow crack was considerably more fibrous than either the crack extension that occurred on initial loading or the fatigue precrack. In an effort to further study the kinetics of the slow crack growth, resistance measurements were employed during the sustained load tests on the 300 M high-strength steel. The variation of resistance with time is presented in Figures 17 and 18 for test temperatures of 300°F and 400°F. At 300°F, an incubation time was observed before slow crack growth was initiated and this incubation time was extremely sensitive to applied stress (see Figure 19). At the 400°F test temperature, no incubation was observed at any applied stress level where delayed failure occurred and slow crack growth was initiated as soon as the load was applied.

The lack of an incubation period is typical of environmentally-induced delayed failure in high-strength steels (4). To evaluate this possibility, sustained load tests were conducted on notch specimens of 300 M at 400°F in an argon atmosphere. The results presented in Figure 20 indicate that the delayed failure at 400°F was completely eliminated when an argon atmosphere was used. The static fatigue in 300 M steel was therefore induced by environmental effects and was presumably due to stress corrosion by the air atmosphere. The strain aging effects previously described with reference to tensile testing were re-evaluated by conducting notch tensile tests on 300 M (600°F temper) in an argon atmosphere. A minimum in notch tensile strength was present at the same test temperatures in both test environments, indicating that the tensile test data were associated with a material phenomenon and not with the external test conditions.

Although the delayed failure is environmentally induced, it is still significant that the temperature region where delayed failure occurred corresponded to the range where strain aging effects were observed. As the crack slowly grows in a constant load test, because of environmental effects, the applied stress is increasing due to the decrease in load carrying area. On this basis, the sustained load test is comparable to a slow strain rate tensile test provided a mechanism, such as stress corrosion, is available to continually produce a slow crack propagation. Strain-aging effects should therefore influence the failure properties in this temperature range just as they decrease the notch tensile strength in conventional tensile tests. The lack of delayed failure in the other materials investigated is in general agreement with their decreased susceptibility for the initiation of slow crack growth and stress corrosion effects (4)*. Additional work is still needed to determine the mechanism whereby mild environments such as air can produce such a rapid deterioration in load carrying ability of a high-strength steel.

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* A possible exception to this statement is the H-11 steel.

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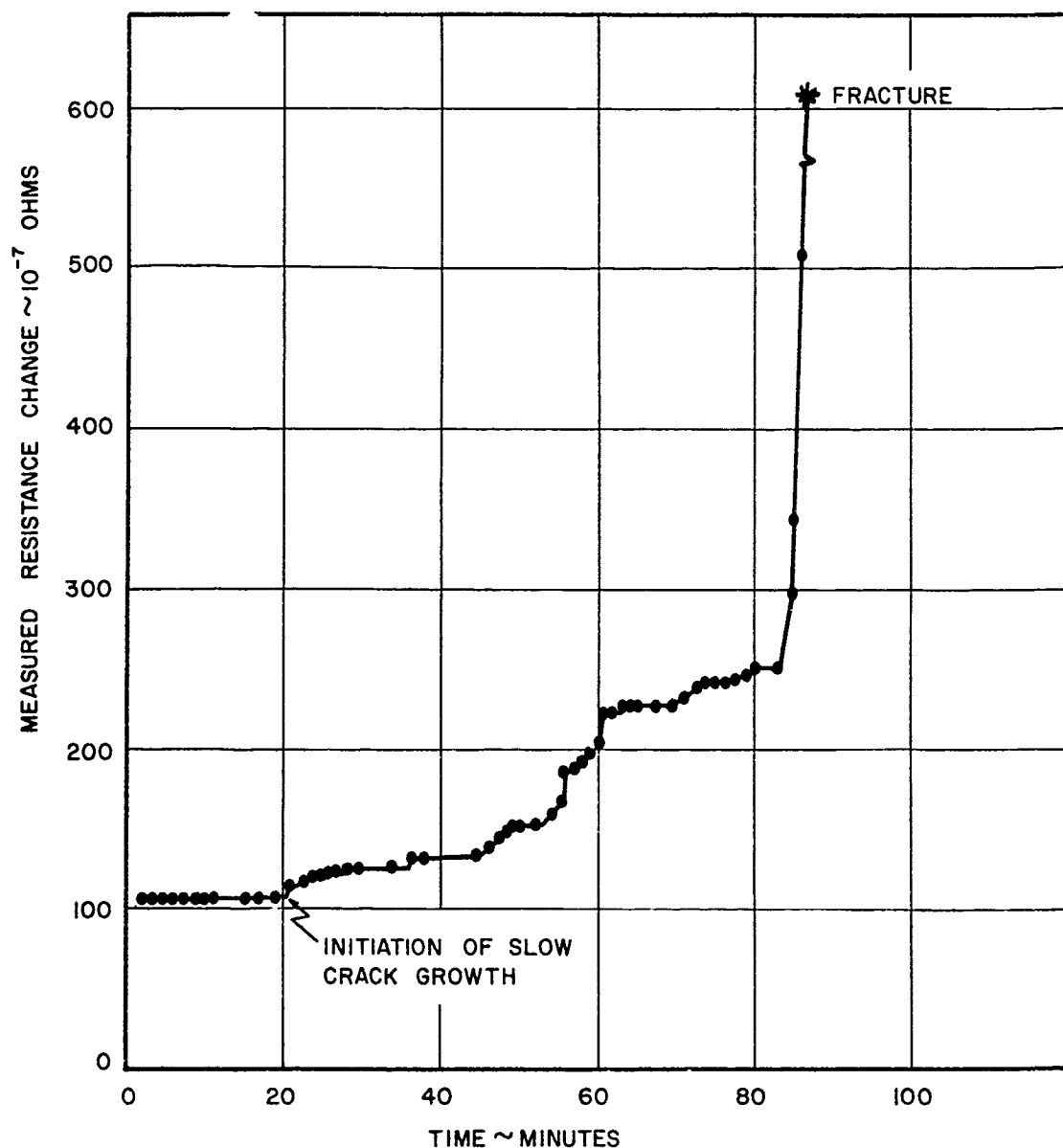
FIG. 17. KINETICS OF CRACK GROWTH AS DETERMINED BY RESISTANCE MEASUREMENTS ON SUSTAINED LOAD TESTS OF NOTCH SPECIMENS OF 300 M STEEL, (600°F TEMPER) TESTED AT 300°F, UNDER AN APPLIED STRESS OF 64,500 PSI.

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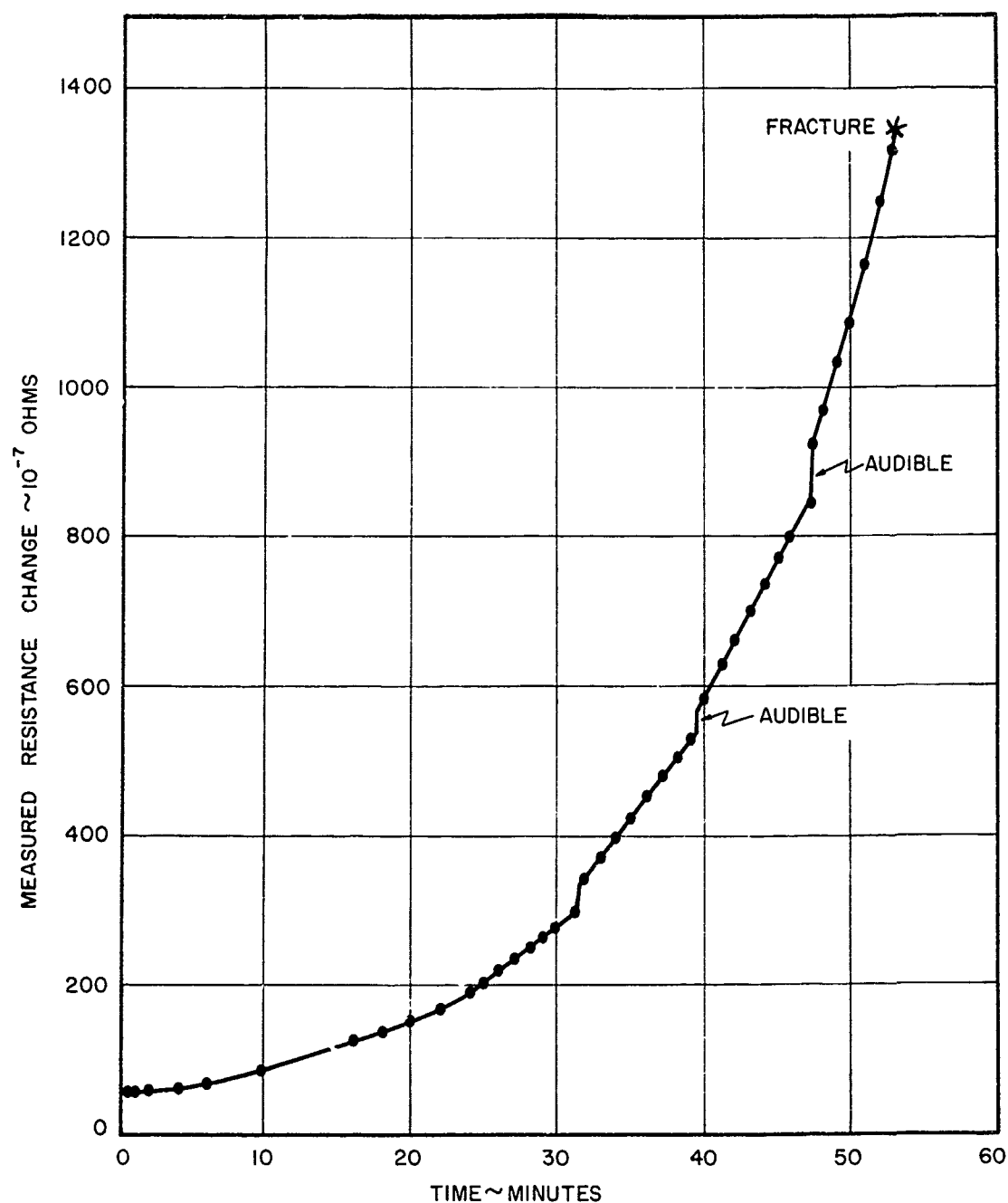
FIG. 18: KINETICS OF CRACK GROWTH AS DETERMINED BY RESISTANCE MEASUREMENTS ON SUSTAINED LOAD TESTS OF NOTCH SPECIMENS OF 300 M STEEL, (600°F TEMPER) TESTED AT 400°F UNDER AN APPLIED STRESS OF 45,750 PSI.
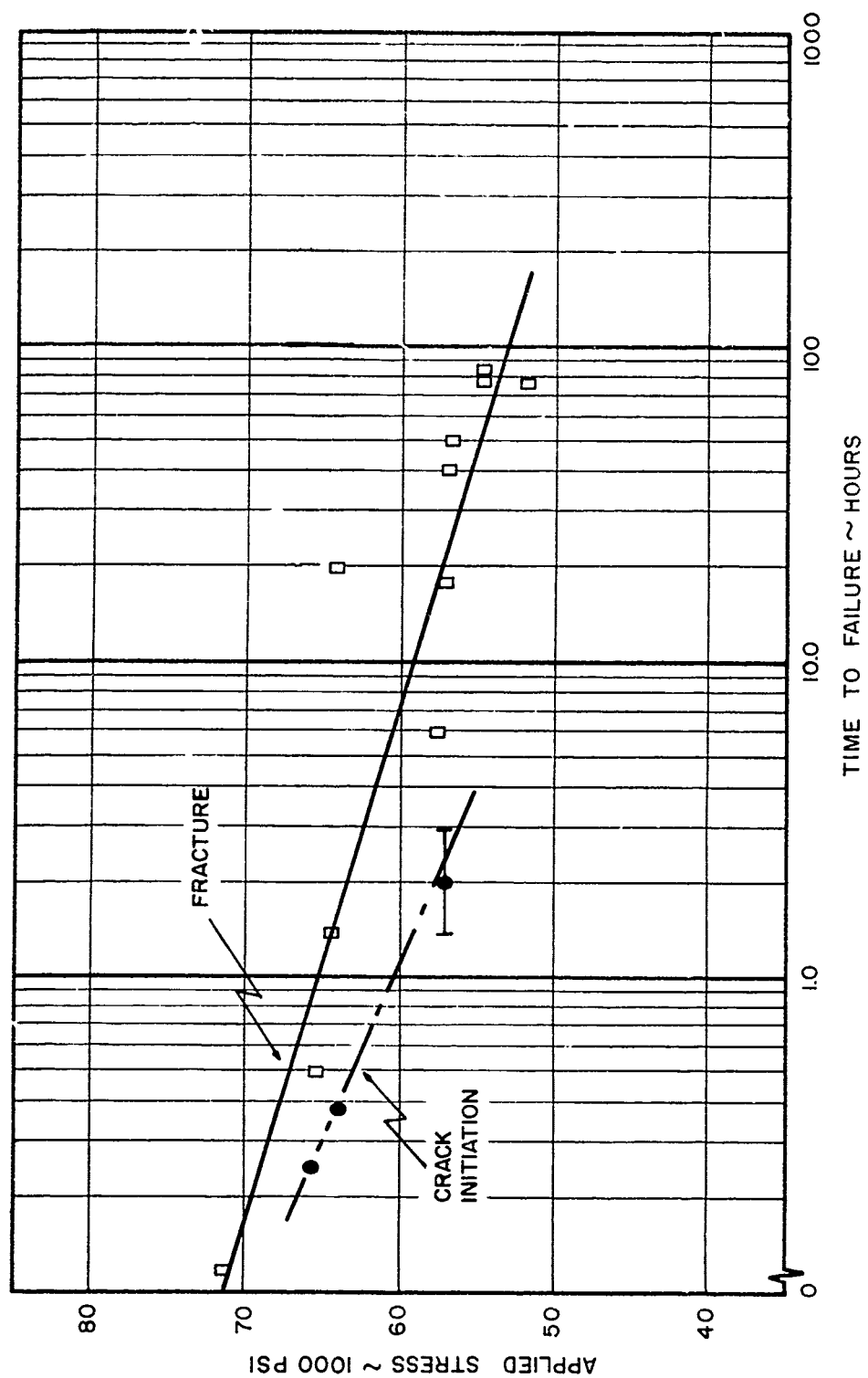
FIG.19: STRESS RUPTURE AND CRACK INITIATION CHARACTERISTICS OF 300 M STEEL, (600°F TEMPER) NOTCH SPECIMENS TESTED AT 300°F IN AN AIR ENVIRONMENT.

28
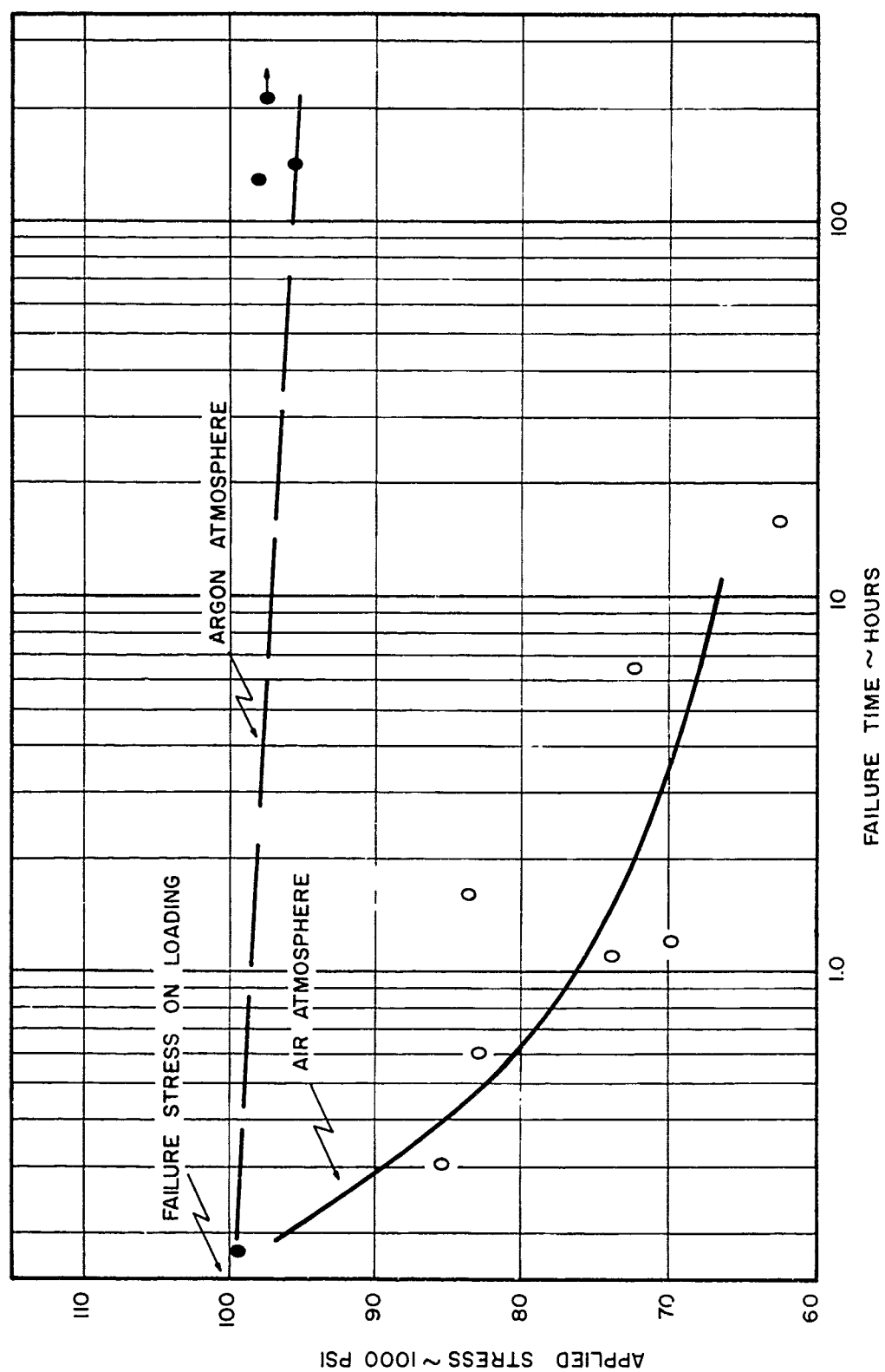
FIG. 20: INFLUENCE OF ENVIRONMENT ON THE DELAYED FAILURE CHARACTERISTICS OF 300 M STEEL, (600°F TEMPER) NOTCH SPECIMENS TESTED AT 400°F.

29

C.   Possible Strain-Aging Mechanisms in High-Strength Steels

Strain aging and "blue brittleness" have been extensively investigated in mild steels and at present the following three factors are believed to be associated with the increase of smooth strength properties which accompany strain aging phenomena:

1)   Short-range ordering about moving dislocations (21, 22),

2)   The dragging of a solute atmosphere with a dislocation line (23, 24),

3)   The formation of a precipitate about a dislocation line (7).

The short range ordering of solute atoms about dislocations has been described in some detail by Schoeck and Seeger (21) and Wilson and Russell (22). The interstitial atom produces a distortion with a symmetry which is different from the symmetry of the lattice. On this basis, the interstitial will take up preferred positions within the lattice when a non-hydrostatic stress field is present. Under certain time-temperature conditions the ordering of the interstitials about the moving dislocation will produce a restraining force because energy is dissipated during the ordering which takes place. This dragging force is not apparent at low dislocation velocities or high-test temperatures where the ordering always reaches thermo-dynamic equilibrium. At high velocities or low temperatures the time is insufficient for redistribution to take place and the restraining force is also absent. An ordering phenomenon of this type produces the well-known Snoek peak in internal friction studies and is characterized by the relatively short times involved, since redistribution only takes place over atomic dimensions. A second type of dragging force which can occur is due to the formation of a Cottrell cloud about a moving dislocation (8, 23). The times required for this strengthening to occur should be approximately 100 times greater than that involved in ordering since the interstitials have to move over greater distances to form the cloud (25).

The role of strain-induced solution and precipitation of carbide particles has been used in both mild (7) and high-strength steels (10, 11) to describe strengthening effects produced by tensile straining, followed by suitable aging. A simple application of precipitation as a mechanism for strain aging which occurs during the test (blue brittleness) is difficult to visualize since it would require the precipitation process to occur on a moving dislocation. Certain internal friction peaks, which involve oscillating dislocations however, have been attributed to very small coherent carbide precipitation (26). In addition, the flow process may be fairly complex in the high-strength steels and may involve intermittent movement of specific dislocations which would allow precipitation effects to become operative. On this basis, the possibility of solution and reprecipitation of carbides in "blue brittle" behavior should not be disregarded.

30

Before briefly discussing the possible significance of each of these three strain aging mechanisms to high-strength steels, it is interesting to evaluate the activation energy for the strain aging behavior. The embrittlement can be expressed in terms of the conventional rate equation:

$$\dot{\varepsilon} = A e^{-Q/RT}$$

where:

$\dot{\varepsilon}$ = strain rate

A = constant

Q = activation energy

R = gas constant

T = absolute temperature

To evaluate the activation energy Q, the logarithm of the crosshead speed was plotted as a function of $1/T$. T was taken as the temperature where the tensile strength was a maximum, or the notch tensile strength was a minimum. The results are presented in Figure 21 for the time-temperature dependency of the notch strength minima and in Figure 22 for the smooth tensile strength maxima. These data indicate that both parameters conform to the conventional rate equation and have an activation energy of approximately 25,000 cal/mole. Due to the difficulty in selecting the exact maxima or minima from the mechanical property data the activation energies can have a spread of approximately ±4000 cal/mole. The data which are available for the "blue brittleness" of mild steels (27) have been plotted in Figure 23. The activation energy for this embrittlement in mild steel is essentially equal to that which occurs in the high-strength steels and strongly suggests that comparable mechanisms are operative.

Although stress-induced ordering of carbon or nitrogen and the drag of Cottrell clouds of interstitials with dislocations are generally accepted as contributing to the spontaneous strain-aging behavior in mild steels, the following results must still be explained before these mechanisms can be applied to the present data on high-strength steels:

1. The temperature region where strain-aging effects occur is higher for steels which have been heat treated at relatively high tempering temperatures.

2. The apparent activation energy for the strain aging effects in both mild steels and high-strength steels is slightly higher than that obtained for the diffusion of carbon or nitrogen in alpha iron.

3. Stress-induced ordering of interstitials (Snoek peak) is not found in internal friction studies of quenched and tempered steels (28).

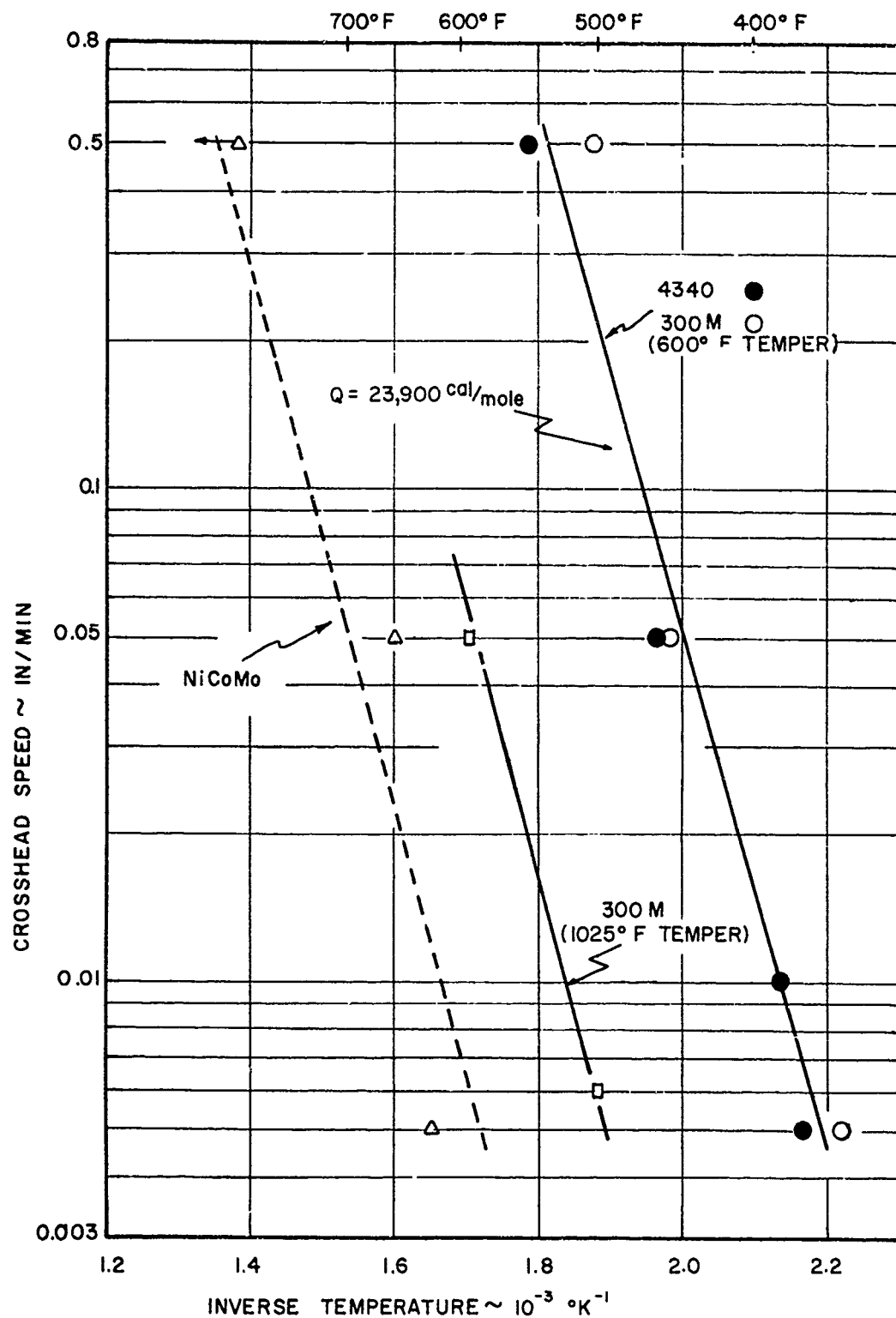31

FIG.21: ARRHENIUS PLOT OF CROSSHEAD SPEED AND TEMPER-
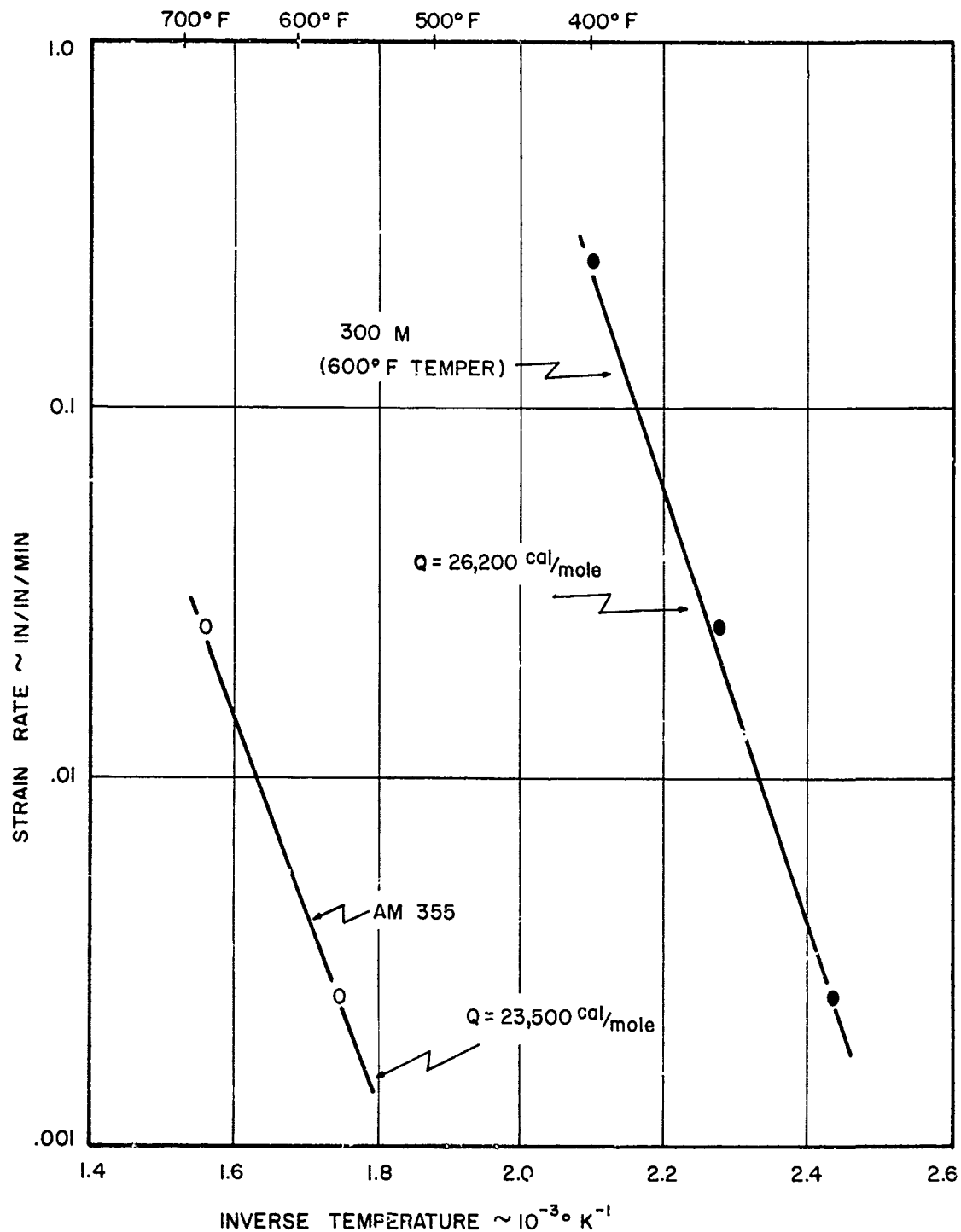ATURE AT WHICH MINIMA IN NOTCH TENSILE
STRENGTH OCCURRED.

FIG. 22: ARRHENIUS PLOT OF STRAIN RATE AND TEMPERATURE AT WHICH MAXIMUM IN SMOOTH TENSILE STRENGTH OCCURRED.
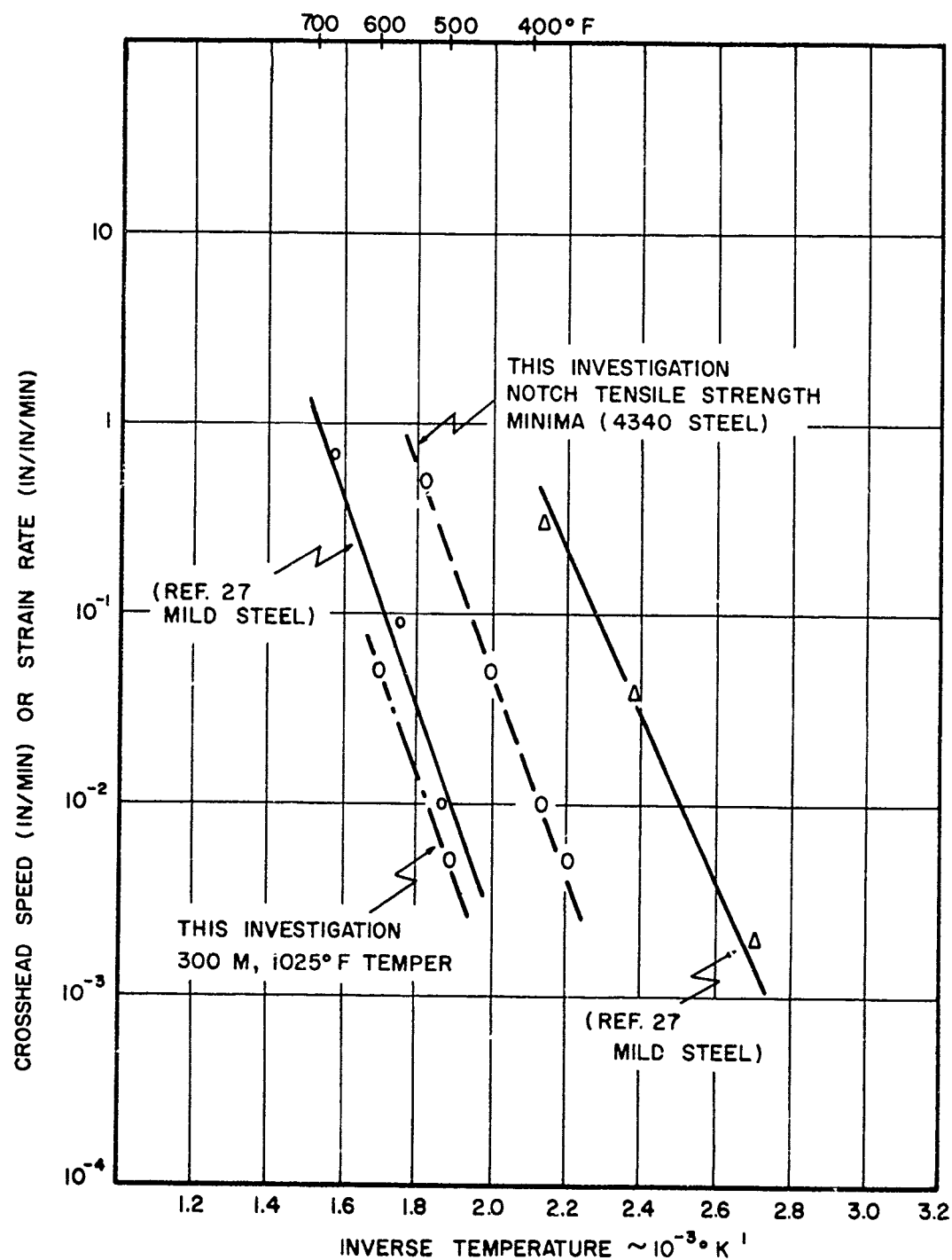
33

FIG. 23: COMPARISON OF DATA OBTAINED FOR "BLUE-BRITTLENESS"
OF MILD STEEL (DUCTILITY MINIMA) WITH NOTCH
TENSILE STRENGTH MINIMA OBTAINED FOR HIGH-
STRENGTH STEELS.

34

Some insight into the poss' ntribution of stress-induced inter-stitial ordering and the Cottreli atmosphere effects can be made by employing a method used by Hahn, Reid, and Gilbert to calculate the effect of ordering on the relationship between strain rate and lower yield stress in mild steel (29). Although these techniques and the assumptions involved are probably over-simplified when applied to high-strength steels, the results can indicate the relative temperature regions where these interstitial effects can be prevalent. The details of the calculations are presented in Appendix I and the results are summarized in Figure 24. The degree of stress-induced ordering is presented in terms of a dimensionless parameter (F) which yields a relative measure of the increase in strength which can be expected by the ordering. The calculations are very dependent on the dislocation density (see equation 2, Appendix I). The results presented in Figure 24 were obtained for two arbitrarily chosen dislocation densities, 10^8 and 10^{10} lines/cm^2. The significant point is that at higher dislocation densities, which would be expected at the lower tempering temperatures, the calculations predict that the strain aging effects should occur at lower test temperatures. These predictions are in qualitative agreement with the observed results on the high-strength steels. There is sufficient uncertainty in the proper selection of the dislocation density so that an accurate appraisal cannot be made of whether a stress-induced ordering of inter-stitials or a gross diffusion of interstitial atmospheres is the primary mechanism. The strengthening due to the drag of interstitial clouds would be approximately two orders of magnitude slower (25) than the stress-induced local ordering pictured in Figure 24. This would raise the temperature at which the strengthening peak would occur by approximately 200°F.

The mechanism of spontaneous strain aging which depends on an inter-action between interstitials and dislocations requires that the activation energy for the strengthening process be equal to the activation energy for the diffusion of the responsible atomic species. Since carbon and/or nitrogen are believed to be responsible for the strain aging effects in the 400°F to 800°F temperature range the activation energy for the strengthening process should be approximately 18,000 to 20,000 cal/mole (8). The ex-perimental data on "blue brittleness" in high-strength steels indicates that this embrittlement is governed by a slightly higher activation energy (approximately 25,000 cal/mole). One possible explanation for this difference is that the actual peaks of tensile strength versus test temperature which are used to determine the activation energy for strain aging are dependent not only on the strengthening process, but are a combined effect of the strength-ening process, superimposed on the normal decrease of strength which accompa-nies an increase of test temperature. This point is illustrated in Figure 25. The sole contribution of the strengthening process is pictured in Figure 25A and this process, in accord with theory (21, 25), can have an activation energy of approximately 20,000 cal/mole. The normal decrease in tensile strength with increasing test temperature is shown in Figure 25B and the combined effects are shown in Figure 25C. The resulting strength peaks are then dependent on the two processes of general softening due to the in-creased test temperature and the strengthening due to interaction effects. As shown qualitatively in Figure 25C, these peaks can produce an apparent activation energy which is greater than that required solely for the strengthening process.
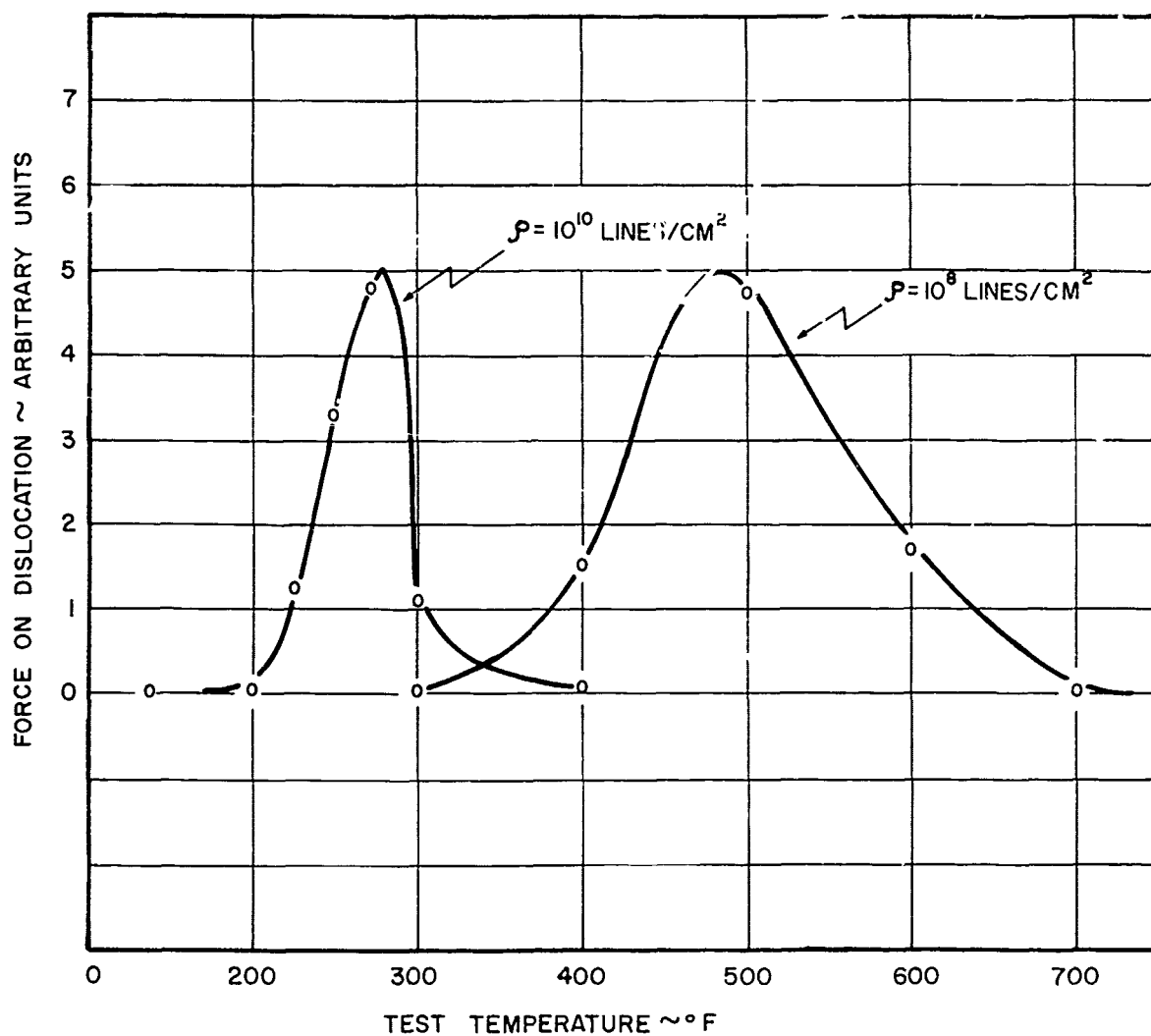
FIG. 24 : CALCULATED FORCE ON DISLOCATION DUE TO
          INTERSTITIAL ORDERING AS A FUNCTION OF
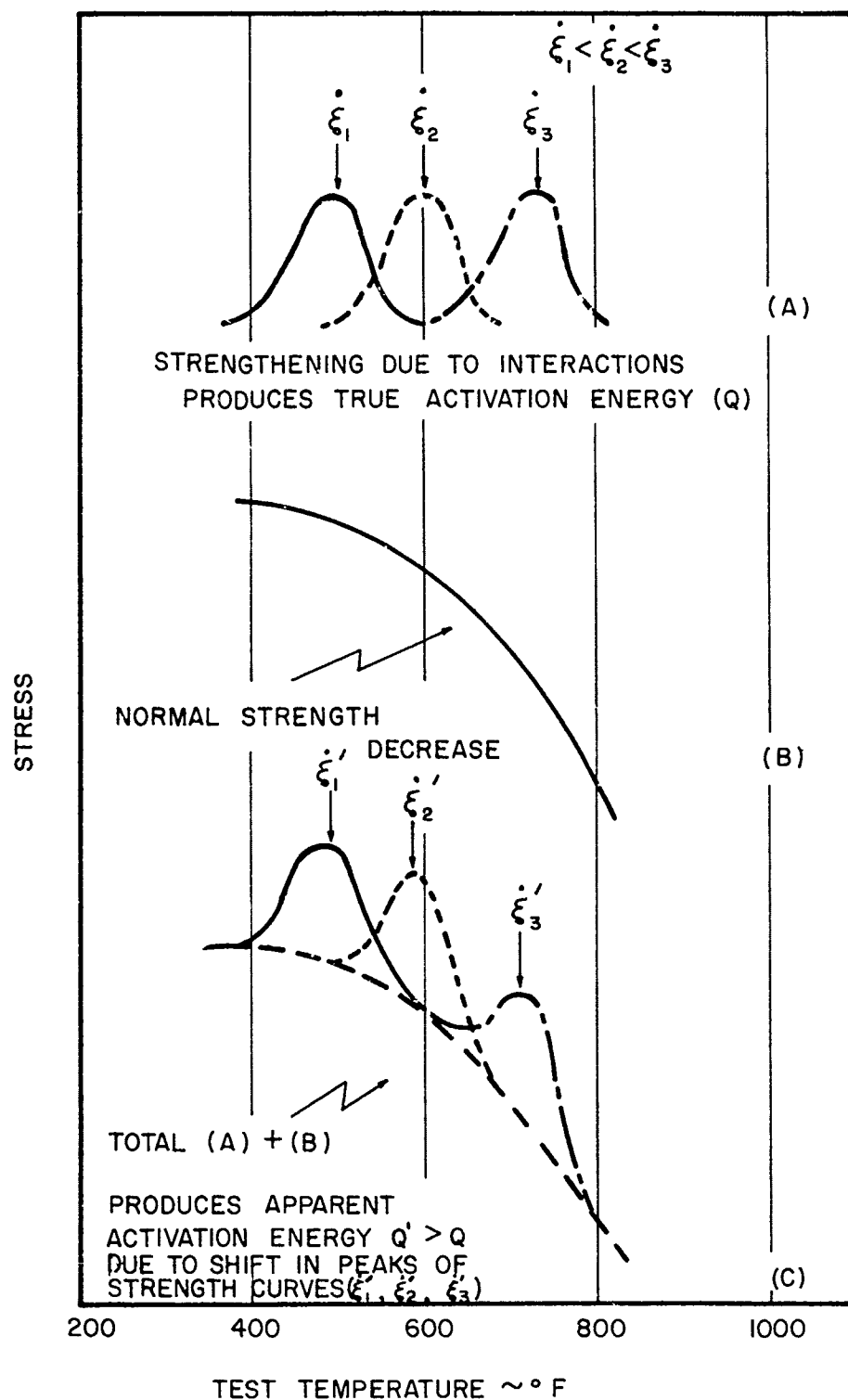   TEMPERATURE.

FIG. 25: SCHEMATIC ILLUSTRATION COMPARING TRUE ACTIVATION
ENERGY OF INTERACTION PROCESS AND APPARENT
ACTIVATION ENERGY OBTAINED FROM TENSILE TEST
DATA.   37

No completely satisfactory explanation is available to explain the fact that internal friction peaks of the Snoek type are not found in quenched and tempered martensitic steels, yet the existence of interactions between interstitials and dislocations are necessary to explain the observed strain-aging effects. Perhaps the presence of certain interaction effects are dependent on a specific degree of plastic flow which occurs in the mechanical tests but which is not normally present in internal friction studies.

Additional support for the mechanism of spontaneous strain aging which involves an interaction of interstitials with dislocations, rather than the formation of a precipitate, is obtained from electron micrographs and pre-stressing experiments. Figure 26 presents the microstructure of 300 M before and after stressing in the temperature range where strain aging takes place. In no case did electron micrography, using replica techniques, indicate any difference in carbide morphology between the specimens which had been stressed to exhibit strain aging and those which had not.

If a precipitate were formed during the tests, in the temperature region where strain-aging effects were present, then this precipitate should also influence room-temperature properties by causing notch embrittlement and an increase in smooth strength. Tests were conducted by prestraining 1" wide, smooth specimens of 300 M, 1.0% at 400°F and allowing the load to remain on the specimen for various times. The specimens were then water quenched, center drilled, and a sharp notch was generated by fatigue precracking. The specimens were tested at room temperature and the results are presented in Figure 27. If an embrittling precipitate were formed by the combined straining and aging treatment at 400°F, then it should be even more effective towards promoting embrittlement at the lower test temperatures. As shown in Figure 27, no evidence of any room temperature embrittlement was present. Similar tests were conducted with smooth specimens of 300 M, where prestraining temperatures of 250°F and 350°F were employed with prestrains of 2.0 and 3.0%. In this series of experiments the specimens were quenched from the prestressing temperature and immediately tested at room temperature to determine if any variations in smooth strength occurred. For the aging times investigated (up to 10 minutes), no significant variation in room temperature tensile strength was obtained. These results indicate that "blue brittleness" in high-strength steels is a unique function of the particular temperature region where sufficient interstitial mobility is present. The strain aging effects are not inherited at other test temperatures and this factor strongly supports a mechanism involving an interaction between interstitials and moving dislocations.

38
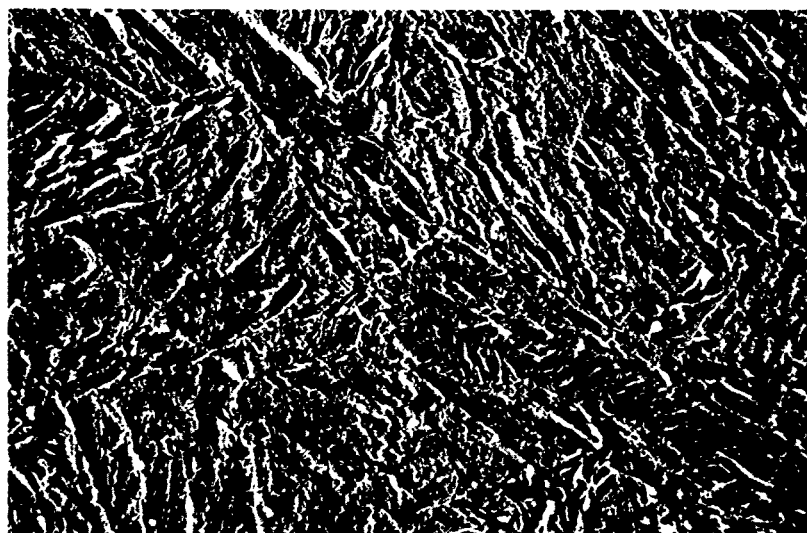
0836

300 M SPECIMEN, STRESSED IN STRAIN AGING REGION, MICROSTRUCTURE OF NECKED REGION
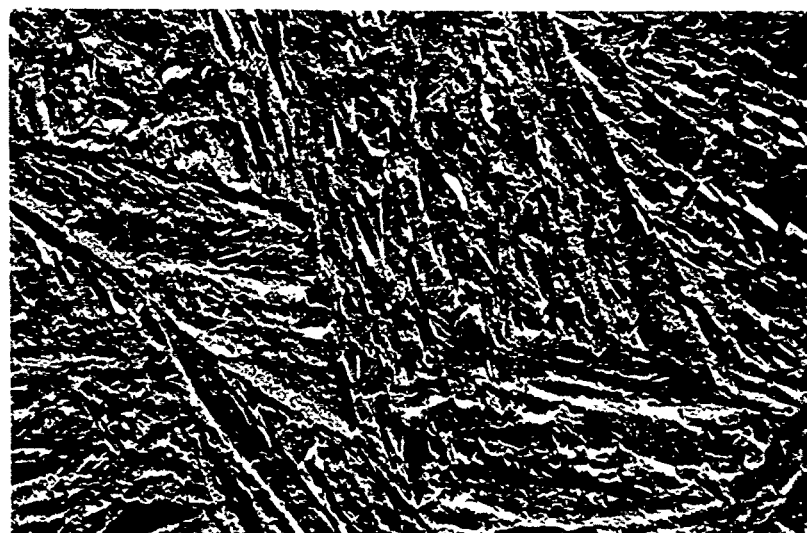
0839

300 M SPECIMEN - UNSTRESSED

FIG. 26: ELECTRON MICROGRAPHS OF 300 M STEEL, TEMPERED AT 600° F, MAG. ~ 10,000 X.
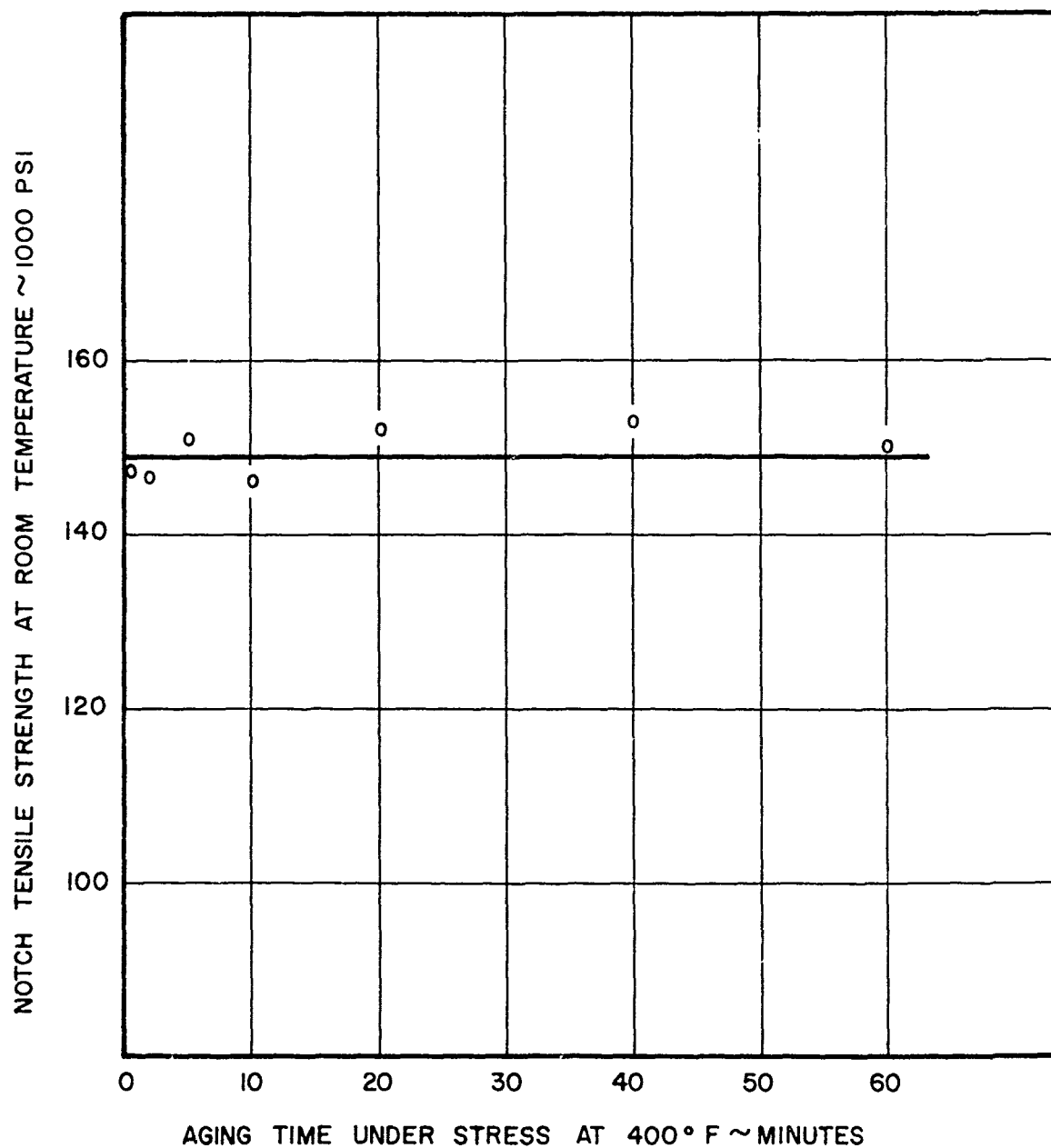
FIG. 27 : INFLUENCE OF PRESTRAINING 1%, AND SIMULTANEOUS
AGING AT 400°F ON THE ROOM TEMPERATURE
NOTCH TENSILE STRENGTH, 300 M STEEL, 600°F
TEMPER.

40

V CONCLUSIONS

High-strength steels can exhibit significant strain aging effects during tensile testing, when tested in the temperature range 300°F to 800°F. The strain aging phenomena are characterized by an increase in smooth tensile strength and a decrease in notch tensile strength. The strain-aging behavior is dependent on strain rate in a manner which is analogous to "blue brittleness" in mild steels. The activation energy for the phenomena is comparable to that shown by "blue brittleness" and is approximately 25,000 cal/mole. The strain aging effects were observed in the low alloy martensitic steels (300 M and 4340), the precipitation-hardening stainless steel (AM 355), and the maraging steel (18% nickel, 7% cobalt, 5% molybdenum). No strain aging was noted in the H-11 hot-work die steel or the 17-7 PH precipitation-hardening stainless steel.

Constant load tests were conducted to determine a possible correlation between the strain aging effects noted in the tensile tests and the susceptibility for delayed failure. An appreciable region of delayed failure was present only in the high-strength 300 M steel tested at 400°F. This delayed failure phenomenon was extremely sensitive to environmental effects and was completely eliminated by testing in an argon atmosphere. Although the strain aging did not initiate failure under sustained load, it was believed to occur during slow environmentally-induced crack propagation and contributes to the decrease in load carrying ability of notch specimens tested at 400°F.

The spontaneous strain aging in high-strength steels which occurred during tensile testing was attributed to an interaction between interstitials and moving dislocations rather than precipitate formation.

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APPENDIX I

Calculation of Ordering Effects Due to Interstitials

Schoeck and Seeger (21) present a functional relationship between the force exerted on a dislocation by interstitial ordering and a dimensionless parameter which involves the relaxation frequency for the interstitial and the speed of the moving dislocation. This relationship which can be expressed as:

$$F\left(\frac{U}{R}\right) = f\left(\frac{\alpha}{\bar{\nu}R}\right) \tag{1}$$

where: F = the force exerted on the moving dislocation expressed in units of U/R,

U = the line energy of the dislocation,

R = the effective radius of the dislocation,

α = the speed of the dislocation, and

$\bar{\nu}$ = frequency factor.

The relationship given in equation (1) has been solved graphically (see eq. 28, Ref. 21) and is presented in Figure 28. The problem of determining the restraining force produced by interstitial ordering on a moving dislocation then resolves itself into determining the values of $\alpha/\bar{\nu}R$ and using Figure 28 to obtain the desired result.

The average speed of the moving dislocations (α) can be related to the strain rate ($\dot{\varepsilon}$) by using the relationship presented by Hahn, Reid, and Gilbert (eq. 1, Ref. 29):

$$\dot{\varepsilon} = 0.5\ bf'\rho\alpha \tag{2}$$

where: $\dot{\varepsilon}$ = strain rate

b = Burgers' vector

f' = constant (approx. 10^{-1})

ρ = dislocation density

α = average dislocation velocity

The average relaxation frequency ($\bar{\nu}$) can be determined from the relationship (21):

$$\bar{\nu} = 1.5\ \frac{D}{b^2} \tag{3}$$

where: D = diffusion coefficient and

        b = Burgers' vector

Using the following constants:

$$b = 2.47 \times 10^{-8} \text{ cm}$$

$$D = 5.2 \times 10^{-4} \ e^{-9000/T} \text{ cm}^2/\text{sec.} \qquad \text{(ref. 8)}$$

$$R = \frac{1.84 \times 10^{-20}}{kT} \text{ cm} \qquad \text{(ref. 21)}$$

the values of F, which have been determined for arbitrarily chosen values of $\rho = 10^8/\text{cm}^2$ and $\rho = 10^{10}/\text{cm}^2$, are summarized in Table III.

46

TABLE III

Calculation of Restraining Force Due to Interstitial Ordering

$\dot{\varepsilon} = 4.1 \times 10^{-4}/\text{sec}$

$\alpha = 3.2 \times 10^{-3}$ cm/sec when $\rho = 10^8$ lines/cm^2

$\alpha = 3.2 \times 10^{-1}$ cm/sec when $\rho = 10^{10}$ lines/cm^2

T (°F)	T (°K)	D (cm^2/sec)	$\bar{\nu}$ (sec^{-1})	R (10^{-7}cm)	$\rho = 10^8$		$\rho = 10^{10}$	
					$\alpha/R\bar{\nu}$	F(υ/R)	$\alpha/R\bar{\nu}$	F(υ/R)
68	293	2.29×10^{-17}	5.62	1.90	3.08×10^3	0	30.8	0
200	366	1.05×10^{-14}	25.8	1.55	8.20×10^2	0	8.20	0
225	380	2.52×10^{-14}	62.0	1.50	353	0	3.53	0.12
250	394	6.31×10^{-14}	155	1.45	146	0	1.46	0.33
275	408	1.29×10^{-13}	317	1.40	73.9	0	0.74	0.48
300	422	2.86×10^{-13}	702	1.35	34.6	0	0.35	0.11
400	478	3.51×10^{-12}	8.65×10^3	1.19	3.19	0.15	0.032	0.04
500	533	2.46×10^{-11}	6.04×10^4	1.07	0.508	0.47	5.08×10^{-3}	0
600	588	1.17×10^{-10}	2.88×10^5	0.97	0.118	0.17	---	0
700	644	4.20×10^{-10}	1.05×10^6	0.89	0.032	0.04	---	0
800	699	1.29×10^{-9}	3.17×10^6	0.82	0.013	0	---	0
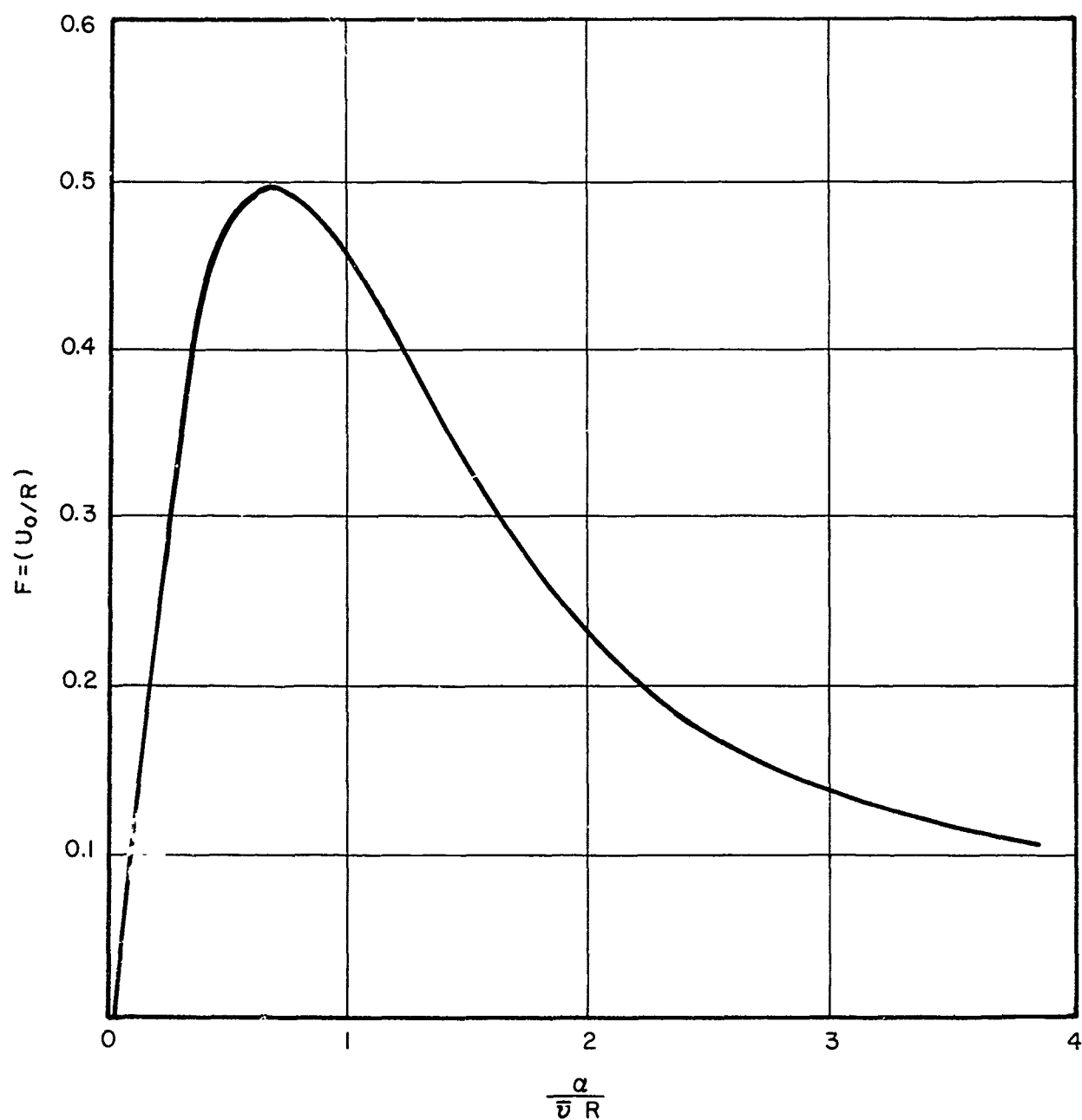
FIG. 28 : THE FORCE TO MOVE A DISLOCATION IN STEADY STATE AS A FUNCTION OF DISLOCATION VELOCITY (α).

48

Aeronautical Systems Division, Dir/Materials
& Processes, Metals & Ceramics Lab, Wright-
Patterson AFB, Ohio.
Rpt No. ASD-TDR-62-968. STRAIN AGING AND
DELAYED FAILURE IN HIGH-STRENGTH STEELS. Fi-
nal report, Nov 62. 48p., incl illus., tables,
44 refs.

Unclassified Report

A study was made of the tendency of several
high-strength steels to exhibit a strain-
aging embrittlement at super-transition tem-
peratures. The 300 M, 4340, AM-355 stainless
and 18% maraging steels were found to exhibit
such an embrittlement.

(over)

1. Tensile Tests
2. Hi-Strength Steels
3. Rupture
4. Dislocations and
   Interstitial
   Interactions
5. Strain-Aging Em-
   brittlement
I. AFSC Proj 7351,
   Task 73521
II. Contract No.
    AF 33(657)-7512
III. Thompson Ramo
     Wooldridge Inc.,
     Cleveland, Ohio
IV. Steigerwald, E.A.,
    Hanna, G.L.
V. Avel fr OTS

VI. In ASTIA collection

Stress rupture tests were conducted on these
steels at the temperatures of the observed
embrittlement to determine the extent of de-
layed failure in each.

The 300 M steel was found to exhibit the
greatest extent of delayed failure. However,
by testing in an argon atmosphere the delayed
failure was completely eliminated, and was
therefore attributed to environment and not
to strain-aging embrittlement.

The kinetics of the strain-aging mechanism
were also studied.